THE ERECTION OF A LIQUID OXYGEN PRODUCING PLANT AND THE REDESIGN OF THIS PLANT TO PRODUCE LIQUID NITROGEN

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by

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Approved:

Date Approved by Chairman June 3, 1948

ACKNOWLEDGMENTS

I wish to express my sincerest thanks to Dr. J. W. Mason for his cooperation in the erection of this plant and to Dr. Gerald A. Rosselot and Dr. Paul Weber of the Experiment Station for making this work possible. I wish also to acknowledge the labors of those persons who assisted in the erection. However, it is with especial gratitude that I acknowledge the unflagging enthusiasm and the unstinting efforts of Dr. W. T. Ziegler on behalf of both this work and this thesis.

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PREFACE

Under the sponsorship of the office of Naval Research of the Navy Department, (contract No. N6-ori-192), a cryogenics laboratory has been established at the State Engineering Experiment Station.

The purpose of this laboratory is to investigate the properties of matter at low temperatures with particular emphasis on the range of 1 to 20°K.

To carry on work at these low temperatures large quantities of liquid nitrogen are necessary as a refrigerant. For the purpose of assuring a plentiful supply of liquid nitrogen the Navy Department made available to the Experiment Station a used liquid oxygen producing plant. The erection and redesign of this plant to produce liquid nitrogen is the subject of this thesis.

The erection, while consisting of the routine matters required to set up and condition used equipment, also provided the time and association necessary to "get the feel" of the plant.

The redesign also, was a gradual evolution in thinking as familiarity with the equipment increased.

While the primary purpose of the plant is to produce liquid nitrogen there is a real possibility that the plant may be used in graduate instruction and research. Flexibility of design is therefore, highly desirable. To this end alterations were planned such that liquid air, oxygen, or nitrogen

may each in turn be produced with changes in valve settings only. Also, an attempt has been made to permit the change of the nitrogen rectification system to another type with a minimum of effort.

Since the plant was originally designed to produce and rectify liquid air to liquid oxygen, only facilities for liquid nitrogen production need by added. Inasmuch as nitrogen is considerably more volatile than oxygen its separation from air is not difficult, and allows certain simplifying assumptions to be made.

The composition of air, given in the table below, is quite constant with respect to time and place[1].

COMPOSITION OF AIR (Volume Per Cent)

N_2	78.03	H_2	1×10^{-2}
O_2	20.99	Ne	1.5×10^{-3}
A	0.933	He	5×10^{-4}
CO_2	0.03	Xe	$.5 \times 10^{-5}$

Since, in this plant, only a small percentage of the total nitrogen present in the liquefied gas is drawn off as product, and no other product is obtained, there is no need to consider air as other than a binary mixture of oxygen and nitrogen. As such it will be assumed to be 21 mole per cent oxygen and 79 mole per cent nitrogen.

Reports of the work by the M. W. Kellog Company for the Office of Scientific Research and Development have been drawn

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[1]Ruhemann, M., The Separation of Gases, Oxford, Clarendon Press, London, 1940, p. 1.

upon heavily as the references show. In no other known source has so much data pertinent to the problems of the liquefaction and separation of air been collected.

CHAPTER I

DESCRIPTION OF THE LIQUID OXYGEN PRODUCING PLANT

The liquid oxygen producing plant furnished by the Navy Department is designated as a Linde Model SM-LO-P270, Portable.[1] This plant is of a type designed and built for the Navy Department by the Linde Air Products Company for use overseas for high purity (99.5 mole per cent) oxygen production.

This particular plant was used in a training school at Portsmouth, Virginia, and arrived minus a number of necessary parts and operating supplies. The plant also showed signs of not having had the proper care. It was not until the Navy sent another plant, this time an unused one, that enough parts and operating supplied were available to operate the plant.

Four units constitute the operating equipment: (1) the compressor, (2) the interchanger, (3) the expansion engine, (4) the storage tank. Each will be described briefly.

The Compressor.[2]--The air to be processed is supplied by a three-stage, six cylinder, single-acting Worthington air compressor, model number V6W3-6/6x7x4¼x5. The compressor is driven

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[1] Producing Plant Operating Manual, Buship I.B. No. 646001 The Linde Air Products Co., Unit of Union Carbon and Carbide Corp., New York, N.Y., 1944, p. I-1. Hereafter this book will be referred to simply as the Operating Manual.

[2] Operating Manual, p. IV-3.

by a 220/440 volt, 3 phase, 100 horsepower, 60 cycle, squirrel cage induction motor.

The air is drawn in from outside the laboratory through two "Air Maze" filters into the compressor where its pressure is raised in three consecutive stages to 300 psia.

The cylinders of the compressor are not jacketed but are provided with cooling fins. However, the air discharged from each stage is cooled in exchangers of the shell and tube type to approximately room temperature. A radiator cooling system is provided for locations where an external source of cooling water is not available.

In normal operation the compressor delivers 15,000 cubic feet (measured at 70°F. and 1 atmosphere pressure) to the interchanger each hour.

The Interchanger[3].--The interchanger is the production unit of the plant. In this unit air is cooled in a series of countercurrent heat exchangers; a portion of the air is partially liquefied in another exchanger and completely liquefied in a condenser coil. The liquefied air is filtered, and throttled into a distillation column to produce 50 pounds of liquid oxygen per hour.

A flow diagram of the plant may be seen in Figure 6.

A distinctive feature of this plant is the method of removal of water vapor, carbon dioxide, and any hydrocarbons

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[3]Operating Manual, p. VIII-3

which the entering air contains. This purifying is accomplished by condensing and freezing rather than by chemical means as is the usual case in oxygen plants.

The first exchanger, called the warm leg, cools the air to a few degrees above the freezing point of water, thereby condensing the major portion of the water vapor. The water is collected in a trap from which it is periodically expelled.

The air next passes to a set of two exchangers, termed the cold leg, in which the air is cooled to well below the freezing point of water. The final traces of water are thereby removed, but the exchanger is subject to plugging with ice. For this reason a parallel set of two exchangers is provided and used when the pressure drop through one set becomes excessive. The frozen set may then be thawed and readied for a return to service without any interruption in the operation schedule.

The same scheme is followed at the point where carbon dioxide is condensed. A parallel exchanger is provided in order that plugging will not necessitate a complete shutdown.

Some of the condensed carbon dioxide is carried from the exchanger as flakes in the liquid air. These particles of carbon dioxide, if left in the air, would collect on the seat of the throttle valve and at other points in the system. Filters of porous CARBORUNDUM are provided to remove any solid carbon dioxide, hydrocarbon, or other foriegn material.

In the geometrical center of the interchanger is located the distillation column. The purified liquid air is throttled

into a tray type rectifying column and the oxygen stripped from it.

The Expansion Engine[4].--The main source of refrigeration in this plant is the expansion engine exhaust. A major portion of the air entering the system is diverted, just prior to the last heat exchanger, to the expander. Air enters the engine cylinder and expands against the piston causing the engine to do work. In working against the piston the air is cooled. Thus, air entering the cylinder at 300 psia. and -160°F. is exhausted at about 20 psia. and -272°F. This cold exhaust, together with cold gas from the rectification column, passes back through the heat exchangers to cool the incoming air.

Whereas in larger plants of this type, the work of the expander is utilized, no attempt to do so is made in this one. The energy of the engine is taken up by a small air compressor, termed a power absorber, which works only against a throttle valve.

The Storage Tank[5].--The storage tank received is not the one which normally attends this plant, but is a smaller, one hundred and fifty gallon tank which was designed for transporting liquid oxygen.

The storage tank is a vacumn jacketed tank. The vacumn is produced by a continously operating mechanical pump connected to the jacket. Additional insulation is provided by an un-

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[4] Operating Manual, p. VI-3

[5] Operating Manual, p. XII-14

determined type of packing in the free space of the jacket.

These precautions lower the heat leak to such a point that less than 1.5 per cent of the liquid oxygen, boiling at -297°F., will evaporate in twenty-four hours.

CHAPTER II

LIQUID OXYGEN PLANT ERECTION

The liquid oxygen producing plant is described as portable. However, the compressor weighs 7,000 pounds and the interchanger 15,000. The weight of these machines and the vibration they produce when operating necessitate good foundations.

The Chemical Engineering Annex, in which the plant is located, was erected on the site of an old fill. Although the fill was known to be at least twelve years old it was still soft. To counteract this condition special bases of heavily reinforced concrete 12 inches thick were poured. Mastic and CELLOTEX strips were used to damp vibration from the bases to the adjacent 6 inch thick concrete floor.

The room in which the plant is located is about 30 feet by 25 feet and has a 14 foot ceiling. An adjoining room which is used for the storage of tools and supplies can be seen in Plate IV.

Plate I shows the compressor in place and in operating condition. The two small pipes at the upper right, leading to the compressor are cooling water intake and discharge lines.

The large box in the right foreground is the motor starter. The starter shown did not accompany the plant but was removed from another similar plant sent by the Navy Department some months after the one pictured here.

On the left of Plate I two large pipes can be seen

PLATE I COMPRESSOR

coming in from the background outside the building; these are air intake lines.

Plate II gives a front view of the interchanger. The majority of the operating valves and gages are located on this front panel. Air is supplied to the interchanger from the compressor at the far right end.

Preparing the interchanger for service was something of a chore. As can be seen in Plate IV the interchanger is large and box-like. In it are housed the various heat exchangers, distillation column, etc. Into the free space between these parts is packed rock wool insulation. Although precautions against the entrance of moisture into the case are incorporated in the design of the unit, at some time previous to its arrival here the insulation had become wet and had been frozen. Freezing had reduced the rock wool to the consistency of sand, making it necessary to remove and replace over a third of the insulation material.

Although the air to the interchanger passes through several traps light hydrocarbons manage to get by into the heat exchangers. In order to ensure that the system be entirely free of oil and grease it was washed out with clean carbon tetrachloride. Approximately sixty gallons were required to wash each exchanger.

The difficulties of introducing into the interchanger the large quantities of carbon tetrachloride necessary were finally overcome by the use of a small plunger pump of the

PLATE II

FRONT VIEW OF THE INTERCHANGER

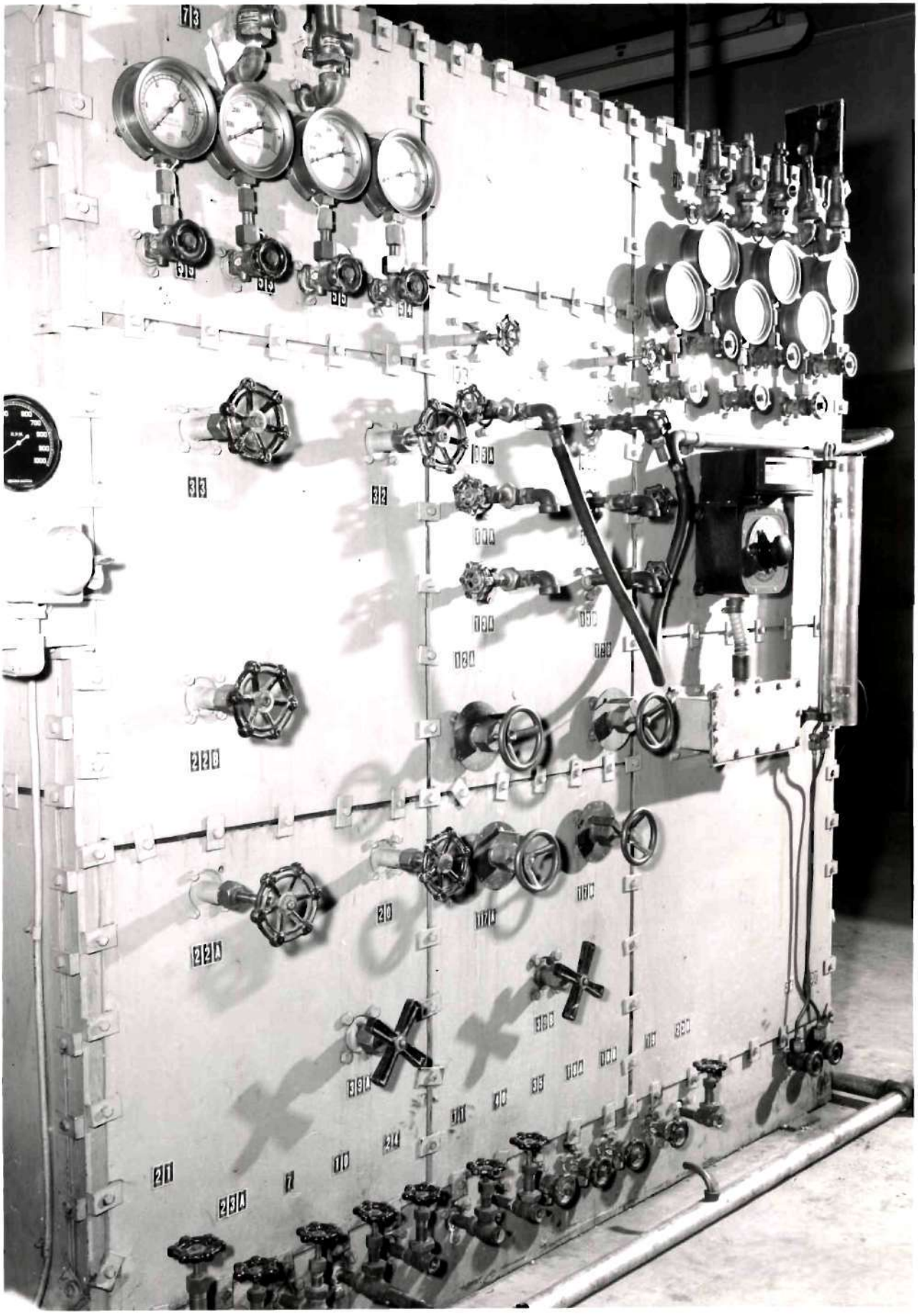
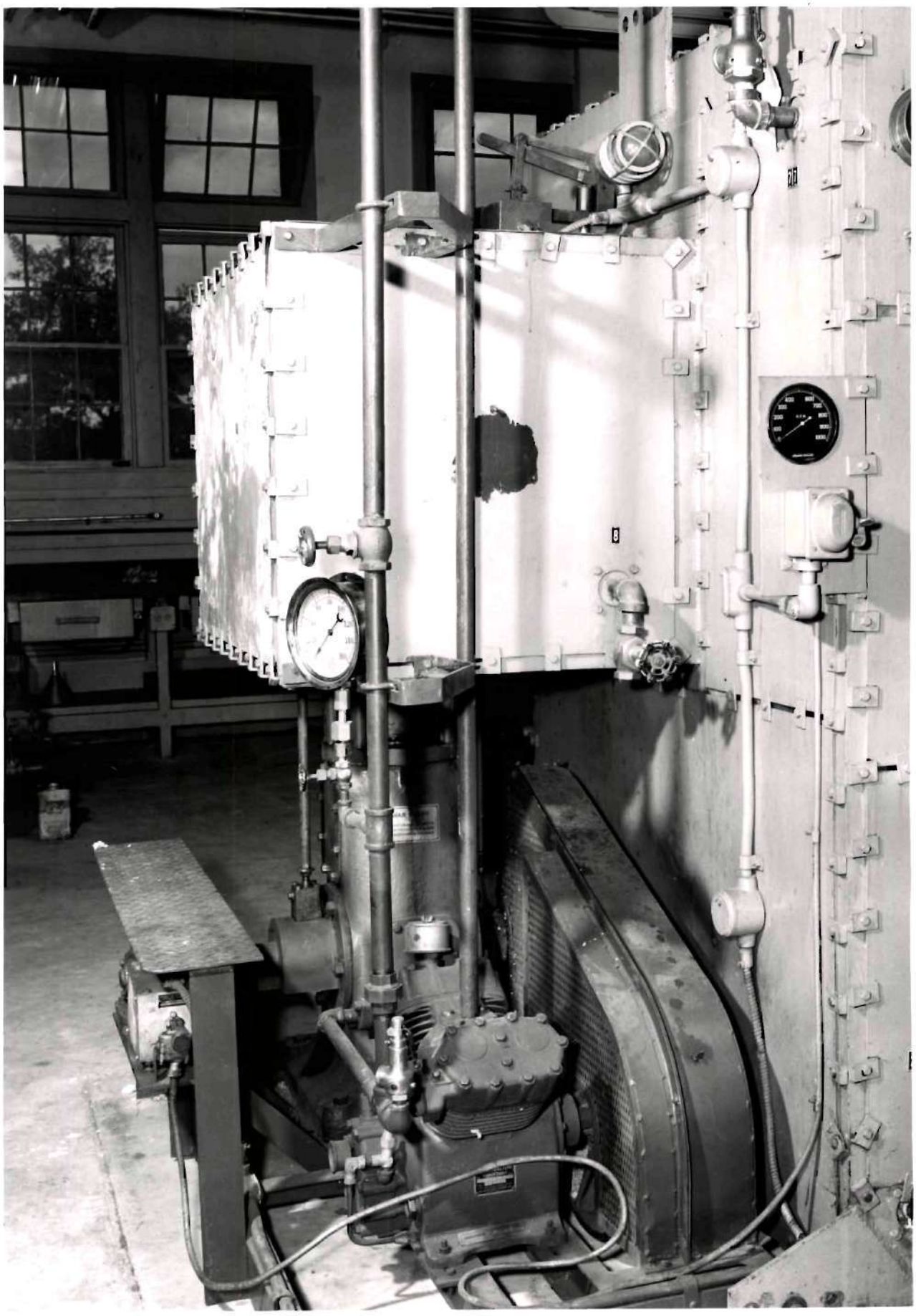


PLATE III EXPANSION ENGINE



type used to dispense *kerosene*.

Plate III is a view of the expansion engine. As can be seen, it is attached to the left end of the interchanger. The upper box-like affair, directly over the engine is an insulated case containing the expansion cylinder. In the center foreground is the power absorber. The black dial on the right, which can also be seen in Plate II, is a tachometer to indicate the expansion engine speed.

The engine shown here is a new one. The one attached to the interchanger on arrival was removed and repaired to be used as a spare.

In Plate IV the 150 gallon storage tank is shown with the front cover removed exposing the operating controls. Until a vacumn pump for the storage tank can be obtained 25 liter thermos cans such as the one immediately to the right of the 150 gallon tank are being used.

The plant has been put into operable condition and produced liquid air. The details of operation are the subject of CHAPTER VIII.

PLATE IV STORAGE TANK

CHAPTER III

JUSTIFICATION, SELECTION, AND INSTALLATION OF A COOLING WATER SYSTEM

During operation the compressor of the liquid nitrogen plant requires a minimum of thirty gallons of water per minute for cooling. The Chemical Engineering Unit Operations Laboratory also requires sizeable volumes of water for operation of its evaporator condenser.

In order to conserve water a cooling tower was installed.

The installation of the tower was justified by a saving of $12.42 per month after payment of depreciation and interest. The Marley Company was low bidder and on the basis of their experience tables their No. 105W tower was purchased.

Professor J. J. Pollard of the Civil Engineering Department calculated the size of the necessary steel supports for the tower. This steel was purchased from Breman Steel Company of Atlanta.

A five horsepower Allis-Chalmers close-coupled, model SS-DH, centrifugal pump was purchased. This pump was installed to circulate water for the cooling system.

The installation of piping, pump, and tower was performed in large part by graduate students working for the Chemical Engineering Department.

Justification of the Installation of a Cooling Tower.--The Producing Plant Operation Manual, BuShip IB. 646001 states

that at least thirty gallons per minute of cooling water necessary for the operation of the air compressor. It was also known that the evaporator condenser in the Unit Operations Laboratory used a large amount of water. The exact amount was unknown but it was in the order of forty gallons per minute.

Seventy-two hours operation per month were predicted for the liquid nitrogen plant and about twenty-four hours per month for the evaporator condenser. The operation of the units is to be scheduled so as not to run concurrently.

$$\frac{72 \text{ Hrs.}}{\text{Mo.}} \times \frac{1800 \text{ gal.}}{\text{Hr.}} \times \frac{\$.16}{1000 \text{ gal.}} = \$20.60$$

$$\frac{24 \text{ Hrs.}}{\text{Mo.}} \times \frac{2400 \text{ gal.}}{\text{Hr.}} \times \frac{\$.16}{1000 \text{ gal.}} = 9.25$$

TOTAL $29.95

If the water were dumped into the sewer the cost would be $29.95 per month.

The investment required for the installation of the cooling system was:

Tower (Marley No. 105W)	$287.00
Tower Base Frame	99.00
Pump and Motor	180.00
Pipe and fittings	150.00
Installation charges	500.00
TOTAL	$1,216.00

Assuming that the equipment was to be amortized over a ten year period and paid for in equal monthly payments, and the interest rate to be 5% per annum on the unpaid balance, the total interest would be $331.00. The total amount to be paid is there-

fore $1,547.00. Each of the 120 monthly payments amounts to $12.90.

The pump requires five horsepower.

$$96 \text{ hrs. operation} \times 5 \text{ H.P.} \times \frac{.746 \text{ K.W.}}{\text{H.P.}} \times \frac{\$.01}{\text{K.W.}} = \$3.63$$

About 3% make up water will be required.

$$\$29.95 \times .03 = \$.90$$

Total monthly charges exclusive of maintenance then approximate $17.43.

The difference of $12.42 per month between cost of cooling without tower and cost of cooling with the tower seemed to justify the installation.

Selection of a Tower.--The selection of a cooling tower demonstrates a lamentable lack of a theoretical basis. No matter what calculations are made, in the end the salesman chooses one on the basis of experience tables.

The heat load of the liquid nitrogen plant was assumed to be equivalent to 100 H.P. 100 H.P. is the rating of compressor motor. It is likely that 100 H.P. is in excess of the actual heat load carried by the water but it supplied a reasonable factor of safety.

$$100 \text{ H.P.} \times 1 \text{ Hr.} \times \frac{2545 \text{ B.T.U.}}{\text{H.P. Hr.}} = 254{,}500 \frac{\text{B.T.U.}}{\text{Hr.}}$$

$$\frac{254{,}500 \text{ B.T.U.}}{\text{Hr.}} \times \frac{1 \text{ Hr.}}{1800 \text{ gal.}} \times \frac{1 \text{ gal.}}{8.33\#} \times \frac{1^{\circ}\text{F. } \#}{\text{B.T.U.}} = 17^{\circ}\text{F.}$$

The calculation above showed that a maximum expected rise in the cooling water temperature to be 17°F.

The thirty year average maximum wet bulb temperature for Atlanta is 76°F.[1] A nine degree approach to the wet bulb temperature by the cooling water was assumed.

The predicted maximum temperature of water onto the tower is 76°+9°+17° = 102°F. A check of the experience tables[2] showed that for water onto the tower at 102°F., and a wet bulb temperature at 76°F. a temperature of 85°F. for water off the tower is probable. The estimated nine degree approach seems safe and the system operable.[3]

For this service bids were requested and a Marley #105W redwood atmospheric cooling tower was selected at a cost of $287.00.

It was decided to place the tower on the roof of the Chemical Engineering building. However, the parapets on the roof of the building are about five feet high and would have cut off the breeze to a large part of the tower. The best way to mount the tower seemed to be to place it on supports resting on the tops of the parapets. Professor Pollard of the Civil Engineering Department calculated the required size of the supports and determined 15.3 lb./ft., 10 inch channels to be

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[1] Perry, J.H., Editor, Chemical Engineers Handbook, 2nd. ed., McGraw-Hill, New York, N.Y., 1941, p. 1109.

[2] Ibid., p. 1112.

[3] See Chapter VIII for information on actual test.

satisfactory. The required length of the channels was 26 feet. The Breman Steel Company returned the low bid of $99.00 for the steel.

Installation of the Tower.--Upon arrival the channels were carried up the stairs of the Chemistry Building and out the window of the back analytical laboratory onto the roof of the Chemical Engineering Building. The tower base frame formed by channels was fixed in place by anchor bolts set in the cast stone cap of the parapet.

The cooling tower was erected on the base frame without difficulty except for one post and three side boards which did not belong with this tower. These boards had to be redrilled in order to be made to fit. Plate V is a view of the cooling tower after installation.

Selection of a Pump for the System.--The selection of a pump resolved itself into the problem of anticipating the required fluid head. Using the rule of thumb that 5 ft./sec. is about the economic velocity for pumping water and the required 30 gpm. flow, results in

$$\frac{30 \text{ gal.}}{\text{min.}} \times \frac{1 \text{ ft.}^3}{7.48 \text{ gal.}} \times \frac{1 \text{ min.}}{300 \text{ ft.}} = .0134 \text{ ft.}^2$$

This area lies between that of $1\frac{1}{4}$" and $1\frac{1}{2}$" pipe. $1\frac{1}{2}$" pipe was selected.

The total fluid head was the sum of the following factors:

1. Height of base of tower above pump (from building plans)	40 ft.
2. Pumping head from base of tower (given by manufacturer)	25 ft.
3. 350 ft. of 1½" pipe with a resistance of 11 ft./100 ft. (Formula of Williams & Hazen, constant 100)	40 ft.
4. 15 elbows at 20 diameters per elbow	4 ft.
5. Other fittings	5 ft.
6. 3 heat exchangers in series	30 ft.
TOTAL FLUID HEAD	144 ft.

Bids were requested for a pump to deliver 30 gpm. against a head of 150 feet. On the basis of cost and delivery date, a 5 horsepower close-coupled Allis-Chalmers ELECTRIFUGAL pump was purchased from John A. Dodd of Atlanta at a cost of $180.00. The pump is at present installed and operating satisfactorily.

Figure 1 is a schematic diagram of the cooling water system. It will be noted that there is a flowmeter and measuring tank in the system. These instruments were the elements of an experiment on the calibration of the flowmeter. They were reinstalled so that their original function would not be disturbed and so that they could also serve to measure the flow of water to the liquid nitrogen plant.

FIGURE I Diagram of cooling water system for Chemical Engineering laboratory and liquid nitrogen plant.

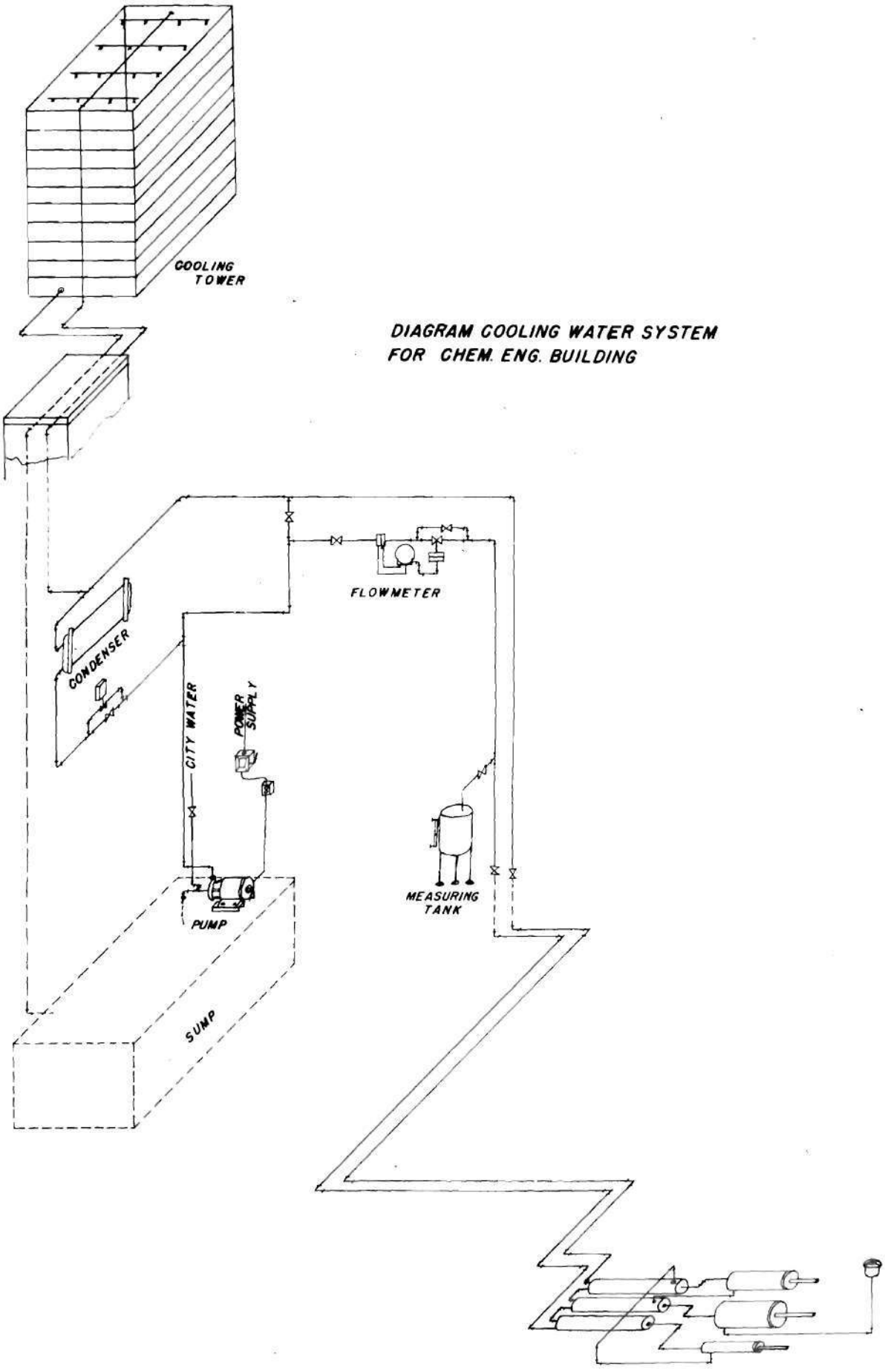
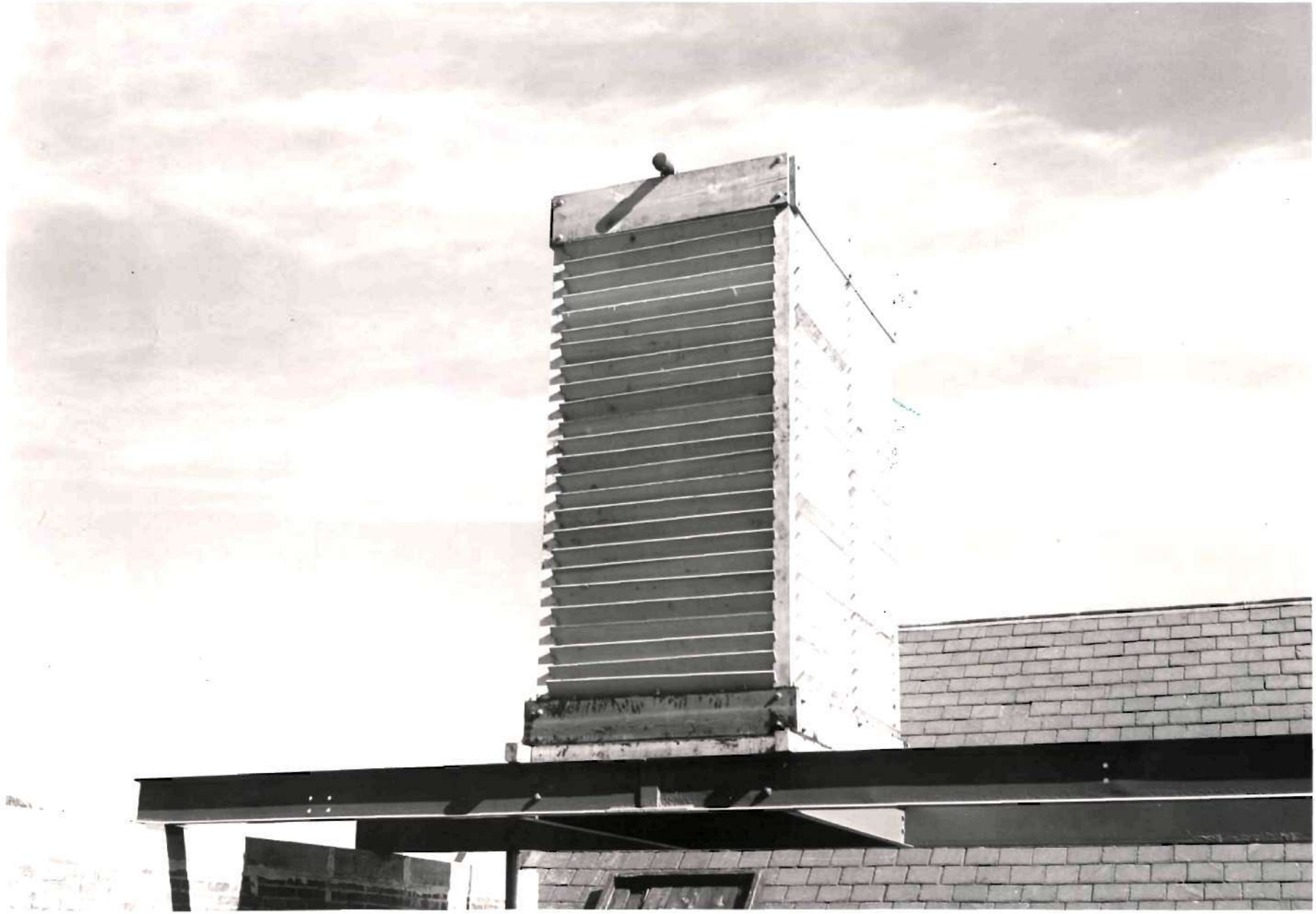


DIAGRAM COOLING WATER SYSTEM
FOR CHEM. ENG. BUILDING

PLATE V COOLING TOWER



CHAPTER IV

LIQUEFACTION PROCESSES

The liquefaction of air depends upon two basic processes. The simpler and older process was first used in a continuous cycle by Carl von Linde in 1895.[1]

The "Linde" cycle utilizies the Joule-Thomson effect to cool air to liquefaction temperatures. However, since the effect is small, particularly at room temperatures, liquefaction cannot occur at once but must be the result of an accumulation of cold. Such an accumulation is accomplished by use of a heat exchanger.

Figure 2 gives a simple schematic diagram of the Linde cycle. Compressed air at room temperature is delivered via the heat exchanger to the throttling valve. After throttling the air is somewhat colder than before and is therefore able to cool incoming gas in the heat exchanger. The incoming gas becomes progressively colder until liquefaction occurs on throttling. After liquefaction begins only the unliquefied portion of the air is returned to cool incoming air and the amount of cooling therefore decreases.

A balance is soon reached such that a small portion of the air is continuously liquefied.

Figure 3 presents a picture of the cycle on the tem-

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[1] Ruhemann, M., The Separation of Gases, Oxford, Clarendon Press, London, England, 1940, p. 114.

perature-entropy diagram after the steady state has been achieved. The numbers in Figures 2 and 3 correspond to identical points in the cycle.

Air is compressed from 1 to 2, cooled isobaricly from 2 to 4, and expanded isenthalpicly from 4 to 5. Point 5 is in the two phase region. The unliquefied air, represented by line 5 to 6, is returned at constant pressure through the exchanger to the atmosphere and, essentially the starting conditions at 8.

The Linde process is simple but requires a relatively large amount of work per unit of liquid obtained.[2]

Several schemes have been devised to increase the efficiency of liquefaction.[3] These include higher pressures, multiple stage expansion, and the use of auxiliary refrigerants.

The Linde cycle is characterized by the isenthalpic expansion and therefore the only reduction of enthalpy takes place on compressing and cooling to room temperature.

A somewhat different approach was made by Georges Claude in 1902. Claude's process derived the major portion of its refrigeration from the use of an engine in which the air was allowed to do work. Since the expansion in a well-insulated engine or turbine approaches an isentropic process this cycle has far greater refrigeration potentialities than the Linde cycle.

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[2]Ruhemann, M., op. cit., p. 125

[3]Dodge, B.F., Chemical Engineering Thermodynamics, McGraw-Hill Book Co., New York, N.Y., 1944, p. 462.

FIGURE 2 - The Linde cycle. Air from the compressor is cooled to room temperature in a water cooled exchanger from 2 to 3; is cooled further in heat exchangers from 3 to 4. At 4 the air is expanded to produce a mixture of liquid and vapor. Unliquefied gas returns through exchangers to atmosphere, cooling incoming air. The liquid air is withdrawn at 6.

FIGURE 3 - The Linde cycle on the temperature entropy diagram. Lines 2-3-4 and 7-8-1 represent lines of constant pressure, while line 4-5 is a line of constant enthalpy. The points correspond to those on Figure 2.

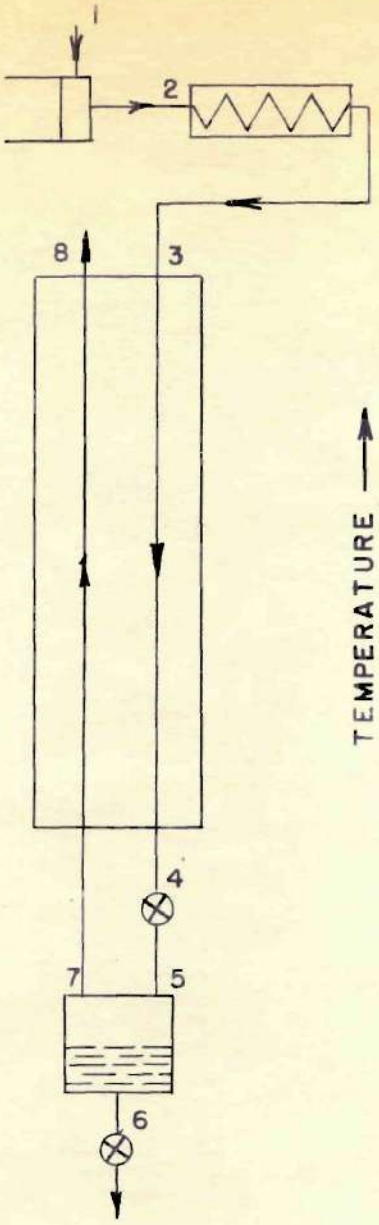

FIGURE 2.

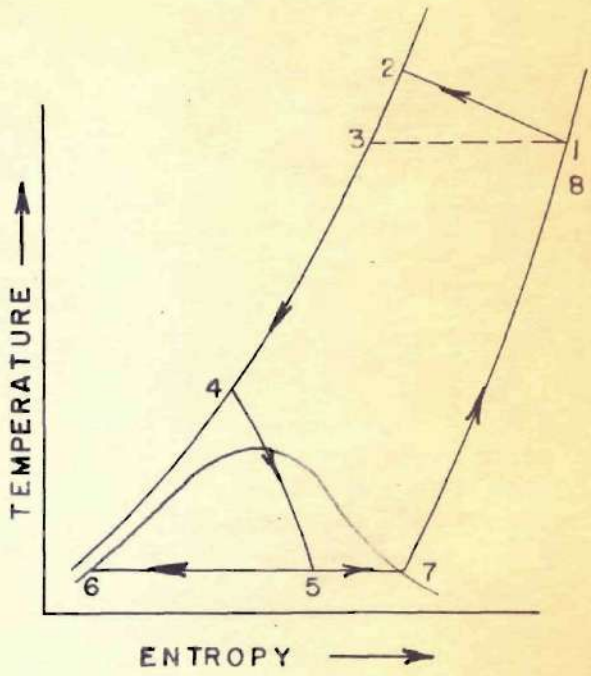

FIGURE 3.

Figure 4 presents the schematic flow diagram for the Claude system. The compressed air is sent through a heat exchanger as before, but then the air stream is divided, and a portion expanded against a piston or turbine. Since exhaust of this engine is very much colder than the intake the exhaust is used to absorb heat from the incoming air.

Due to mechanical difficulties it is not feasible to produce liquid in the engine and liquefaction again depends upon the Joule-Thomson expansion.

Figure 4 shows an additional heat exchanger, II, into which the engine exhaust passes. The high pressure air normally condenses in this exchanger, and hence it is often called the liquefier.

Figure 5 indicates the Claude cycle on the temperature-entropy diagram. Compression takes place from 1 to 2. Heat exchanger I cools the entire stream from 2 to 3. At 3, part of the gas is diverted to the expander and follows the part 3 to 8. The remainder of the stream is cooled in the liquefier to point 4, at which point it is throttled along a constant enthalpy line to point 5. The liquid is collected in the receiver and the flashed vapor returns to the atmosphere and point 9 through heat exchangers II and I, respectively.

This cycle is of particular interest to this thesis since it is essentially the one used in the Linde Liquid Oxygen Producing Plant, Portable.[4]

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[4]A more detailed analysis of the cycle used will be found in Chapter VI.

FIGURE 4 - The Claude cycle.

Compressed air is cooled in heat exchangers to 3. Part of the air is expanded in an engine and the exhaust used to cool unexpanded air in exchanger II. At 4 the rest of the air is expanded isenthalpicly and the unliquefied portion is returned through exchangers II and I. Liquid is drawn off at 6.

FIGURE 5 - The Claude cycle on the temperature-entropy diagram.

Line 3 to 8 represents air expanded in work engine.

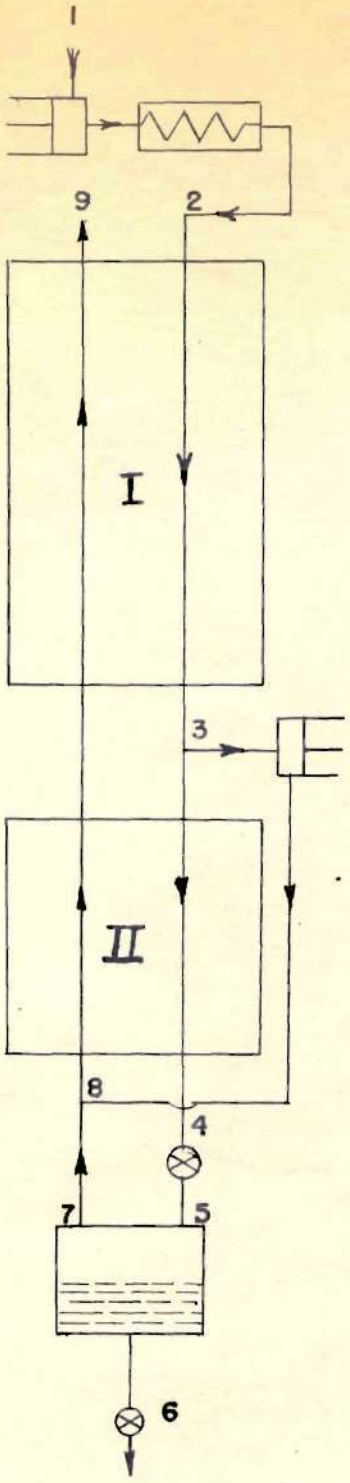

FIGURE 4.

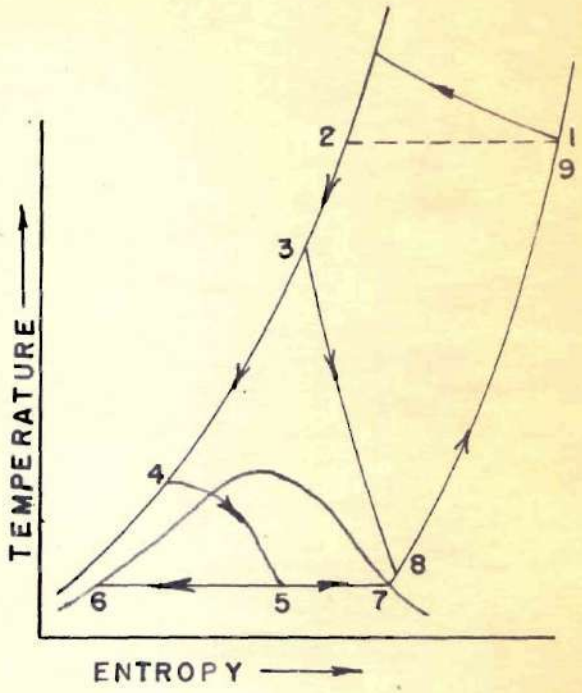

FIGURE 5.

FIGURE 6 - Flow diagram of liquid oxygen plant.

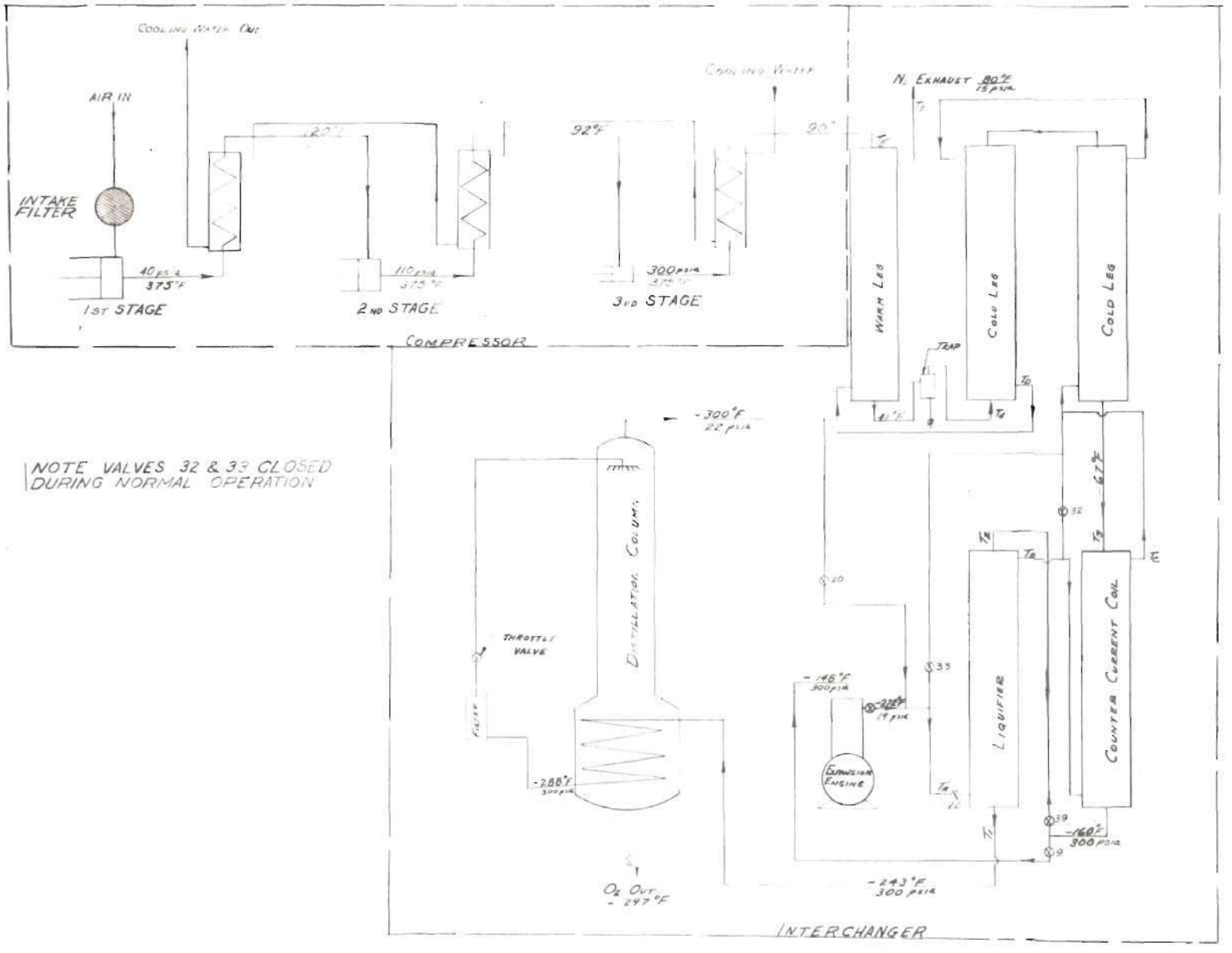
COOLING WATER OUT
AIR IN
INTAKE FILTER
40 psia 375°F
1ST STAGE
110 psia 375°F
2ND STAGE
300 psia 375°F
3RD STAGE
COMPRESSOR
COOLING WATER IN
92°F
90°
N. EXHAUST 80°F 15 psia
WARM LEG
COLD LEG
COLD LEG
TRAP
-300°F 22 psia
NOTE VALVES 32 & 33 CLOSED DURING NORMAL OPERATION
DISTILLATION COLUMN
THROTTLE VALVE
FILTER
-288°F 300 psia
O2 OUT -297°F
-148°F 300 psia
19 psia
EXPANSION ENGINE
LIQUIFIER
COUNTER CURRENT COIL
-160°F 300 psia
-243°F 300 psia
INTERCHANGER

Figure 6 is a flow diagram of this plant. The top pressure used in this cycle is 500 psia. The heat of compression is removed after each compression stage in water cooled exchangers. The entire air stream is cooled in the warm leg, cold leg and countercurrent coil. At valves 9 and 39 the air stream is divided. The major portion of the air flows to the expansion engine and only the air to be liquefied goes through the liquefier and into the column.

The air from the liquefier is a mixture of liquid and vapor, and as such gives up its heat of condensation in the reboiler of the still. This heat is necessary for rectification of the air.

The Claude cycle is normally operated at a top pressure of 40 atmospheres.[5]

This plant, however, while operating on the same principle uses a top pressure of only 20 atmospheres. In terms of efficiency a lower pressure probably indicates higher power consumption per unit of liquid produced. However, the unit was designed to have some degree of portability which would have been difficult to retain with the heavier equipment necessary for higher pressures. Power consumption was undoubtably of secondary importance.

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[5]Ruhemann, M., op. cit., p. 126

CHAPTER V

AIR SEPARATION

In commercial installations air is separated into its component fractions as means of obtaining the pure gases. Historically oxygen was the desired gas. Gradually uses developed for the nitrogen and argon. In recent years commercial attempts have been made to separate xenon and krypton and even helium has been extracted from the air.[1]

The methods used to separate air, at least for the major constituents, follow directly the normal proceedure for separating liquid mixtures. Rectification of liquid air, while requiring some special techniques and equipment, is based upon the same principles as rectification of other more commonplace mixtures, e.g. alcohol and water.

In Figure 9 is shown the original scheme devised by Linde for separating oxygen from the air. The feed was to the top of the column. Heat for reboil was supplied by feed from the liquefier in a coil in the kettle. There is no condenser overhead; therefore there can be no reflux. Since liquid oxygen of high purity (99.5+ per cent) is collected in the kettle, the gas leaving the top of the column contains essentially all of the nitrogen.

The feed to the column is, in the general case, a mix-

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[1] Ruhemann, M., op. cit., p. 2.

FIGURE 7 - The original Linde column for producing liquid oxygen.

The cold high pressure air is expanded at valve "A" to form a mixture of liquid and vapor. The liquid is rectified in the tray column. The liquid oxygen is withdrawn from the bottom at "B". The nitrogen and unrecovered oxygen leave the top and pass back through the heat exchanger.

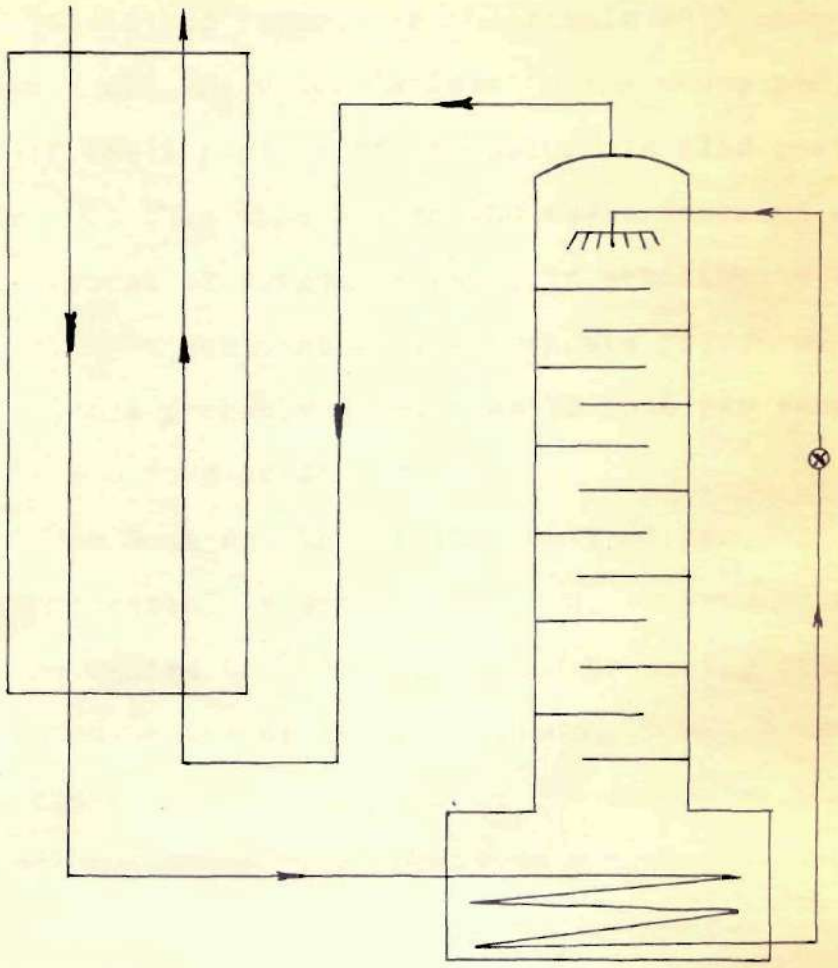

FIGURE 7.

at a temperature of -290 F. The boiling point of oxygen at 1

ture of liquid and vapor. In the simple stripping column shown in Figure 7 all the vapor is lost to the waste gas. Vapor boiled off the top plate of the column is also part of the return gas. In this type column the waste contains as a minimum the amount of oxygen in vapor in equilibrium with liquid air[2] (6.25 mole per cent at 1 atmosphere pressure) and in actual columns probably as much as 10 mole per cent of the oxygen in the feed is lost.[3]

In as much as air contains only 21 per cent oxygen, 10 per cent represents a substantial loss of production.

The desire to increase oxygen producing capacity led to the introduction of the full column, i.e., a rectifying section above the feed as well as the stripping section below. A full column, however, presupposes a condenser for the more volatile component and a condenser presupposes a cooling medium of lower temperature than the nitrogen which boils at -320.5°F.

In as much as no cooling medium of lower temperature than nitrogen is available it is necessary to resort to a special technique.

The pressure of the column is raised from 1 atmosphere to 5 atmospheres. At this pressure nitrogen can be condensed at a temperature of -290°F. The boiling point of oxygen at 1

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[2]Dodge, B.F., op. cit., p. 478

[3]See Appendix A for calculations leading to this result.

atmosphere pressure is -297.5°F. Therefore, it is possible to condense nitrogen at 5 atmospheres pressure with liquid oxygen at 1 atmosphere.

Figure 8 shows how this is carried out. The oxygen is withdrawn from the still pot of the pressurized column and throttled into the condenser at the top of the column and allowed to boil at atmospheric pressure.

The device of raising the boiling point of nitrogen and condensing it with liquid oxygen suffices to provide reflux for the column, but, as is well known, it is not possible to obtain two pure components from a complex misture in a single distillation column. As the demand for pure nitrogen increased, therefore, it was necessary to further rectify the oxygen withdrawn from the stillpot. To provide reflux, nitrogen is withdrawn and introduced above the oxygen feed. The end result is a complete double column as depicted in Figure 9.

The liquid oxygen producing plant furnished by the Navy Department is equipped with a simple stripping column. In order to produce the liquid nitrogen desired it will be necessary to provide some modifications similar to those described above.

A column equipped with a condenser as shown in Figure 8 is adequate to produce liquid nitrogen of any desired purity.

FIGURE 8 - Single column with nitrogen condenser.

This type of column can produce either pure oxygen or nitrogen but not both. The nitrogen is condensed by the bottom product boiling in the condenser at a lower pressure than that of the column. The main feed is throttled at "A". The bottom product is expanded into the condenser through "B". Liquid nitrogen may be drawn off at "C".

FIGURE 9 - A complete double column.

Both pure oxygen and pure nitrogen can be produced in this type column.

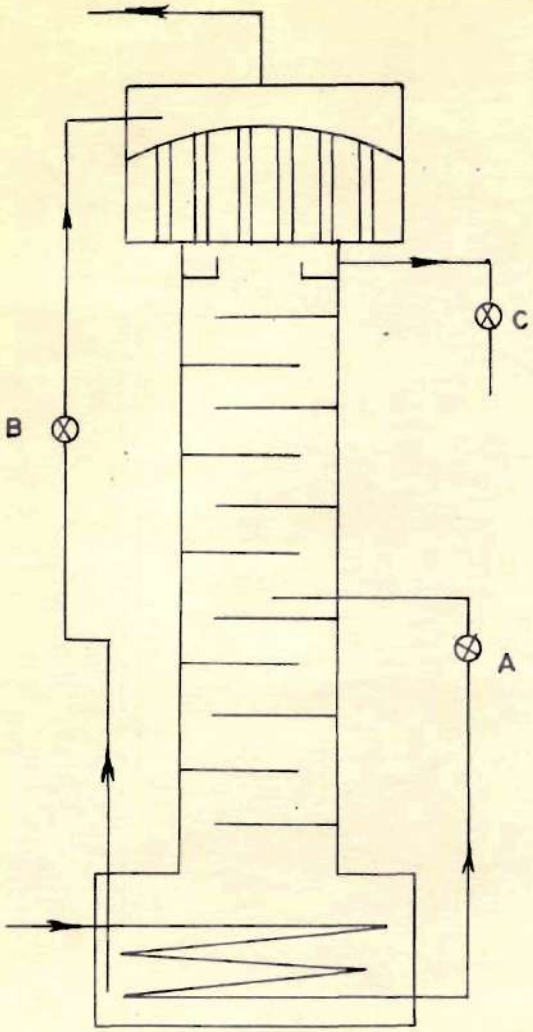

FIGURE 8.

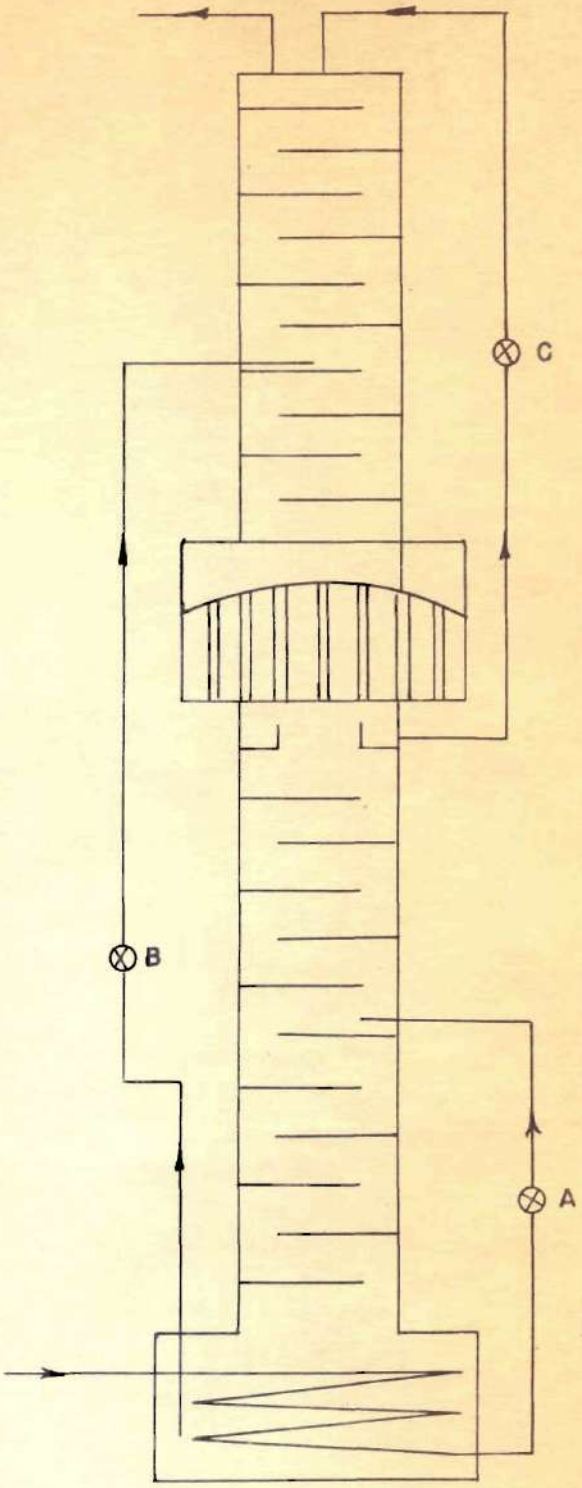

FIGURE 9.

CHAPTER VI

PRELIMINARY THERMAL ANALYSIS OF PLANT

The liquid oxygen producing plant was supplied by the Navy Department with operating instructions and information only. Almost no technical data were included in the Operating Manual. However, with the aid of performance data from the Operating Manual and thermodynamic charts[1,2] the following analysis has been made of the plant as supplied:

Assuming steady operation under normal conditions, a material balance was made in order to show air distribution in the system. Using the results of the material balance and the known normal operating temperatures of the heat exchangers, an enthalpy balance was computed for each section of the plant.

The effect of water vapor and carbon dioxide in the initial air was calculated and included in the enthalpy balance.

The first step in the analysis was to determine the distribution of air in the system.

Figure 6 is a flow diagram of this plant as built.

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[1]Lobo, W.E., O.S.R.D. Report, No. 4206, Technical Data Pertaining to Air, Its Liquefaction and Distillation, M.W. Kellog Co., 1944.

[2]Lobo, W.E. and Williams, Brymer, O.S.R.D. Report, No. 3768, Liquid Air Fractionation, M.W. Kellog Co., 1944. These two works have been of especial aid in the preparation of this thesis and have been drawn upon heavily.

This Figure also gives temperatures and pressures of the gas streams at various points. Referring to Figure 6, the compressed air passes successively through the heat exchangers designated as, the warm leg, the cold leg, and the countercurrent coil. After leaving the countercurrent coil the air is divided and part goes to the expansion engine and part to the column by way of the liquefier and condensing coil.

In the expansion engine the air does work by reason of its pressure and at the expense of its internal energy. In expanding the air cools from -148°F. to about -272°F. The cold exhaust passes countercurrently through the liquefier and other heat exchangers to the atmosphere.

The second high pressure air stream from the countercurrent coil goes to the liquefier where it is partially condensed. The liquid mixture from the liquefier enters the condenser coil in the kettle where condensation is completed and some degree of subcooling takes place. The subcooled liquid is filtered and throttled into the column.

Material balance.--The liquid air feed to the column contains 21 mole per cent oxygen. Of the entering oxygen an assumed 10 mole per cent is lost to the column waste gas,[3] so that the oxygen produced is derived from the remaining 11 mole per cent.

---

[3] See Appendix A for calculation of amount of oxygen lost.

$$\frac{50 \text{ lb. } O_2}{\text{hr.}}^{4} \times \frac{29 \text{ lb. air}}{\text{mole air}} \times \frac{1 \text{ mole air}}{32 \text{ lb.}(.21 - .10)O_2} = 412 \frac{\text{lbs. air}}{\text{hr.}}$$

are fed into the column.

The compressor is rated to deliver 15,000 cubic feet per hour of air measured at 70°F. and 1 atmosphere pressure. This amounts to 1,123 pounds of air per hour delivered to the interchanger. Therefore, the 711 pounds of air not processed in the column are sent to the expansion engine.

Enthalpy balance of Column.--A consideration of the heat effects in the column yields the following equation:

$$H_F + H_L = H_P + H_E$$

where H_F = Wt. of feed/hr. x enthalpy of feed.

H_L = Total heat leak/hr.

H_P = Total heat out in product/hr.

H_E = Total heat out in effluent gas/hr.

Thermodynamic charts[1] are available for air, oxygen-nitrogen mixtures and for the pure components, having the common base point of h = 0 at -460°F. and 0 mm Hg pressure.

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[4]The Operations Manual states that the plant normally produces liquid oxygen equivalent to between 600 to 700 cubic feet per hour measured at 70°F. and atmospheric pressure. Examination of log sheets however reveals that the production was mostly about 600 cubic feet per hour. Six hundred cubic feet is roughly equivalent to 50 pounds of oxygen and as the weight is more convenient to use, 50 pounds is chosen to represent the production of the plant hereinafter.

The same base is used here:

H_P = weight of oxygen withdrawn x enthalpy of liquid oxygen
= 50 lb./hr. x -56.5 B.T.U./lb.
= 2825 B.T.U./hr.

H_E = (412 - 50)lb./hr. x 37 B.T.U./lb.
= 13394 B.T.U./hr.

H_L = 412 lb./hr. x 2 B.T.U./lb. of feed[5,6]
= 824 B.T.U./hr.

By difference H_F = 9,745 B.T.U./hr.

then the enthalpy of feed = $\frac{9,745 \text{ B.T.U./hr.}}{412 \text{ lb./hr.}} = 23.6 \frac{\text{B.T.U.}}{\text{lb.}}$

A check of the total heat load and refrigeration potentialities of the plant other than the column should provide a rough check on the above heat balance.

The total sensible heat load on the system was determined to be 87,672 B.T.U. per hour.[7] The total refrigeration potentialities were found to be 94,800 B.T.U. per hour.

The difference of 7,128 B.T.U. per hour is available for liquefying the 412 pounds of air in the liquefier.

$$\frac{7,128 \text{ B.T.U.}}{\text{hr.}} \times \frac{1 \text{ hr.}}{412 \text{ lbs. air}} = 17.3 \frac{\text{B.T.U.}}{\text{lb.}}$$

---

[5]Dodge, B.F., op. cit., p. 479, has assumed the heat leak in an liquid air plant to be 90 B.T.U. per pound mole of air entering.

[6]Lobo, W.E., and Williams, Brymer, op. cit., p. 17, have found in systems not very different in size, the heat leak into the column to be about 2 B.T.U. per pound. Since the letter more closely approximates the present conditions it is used and assumed to be 20 per cent of the total heat leak into the plant.

[7]See Appendix B for detailed enthalpy balance of the plant.

The enthalpy of saturated air at 300 psia. is 40 B.T.U. per pound and if the enthalpy available for liquefaction is removed the final enthalpy of air leaving the liquefier results. This value of 22.7 B.T.U. per pound compares very well with the corresponding value of 23.6 B.T.U. per pound obtained by the column balance.

These results will be useful in the calculations for a nitrogen column in CHAPTER VII.

CHAPTER VII

DESIGN OF A LIQUID NITROGEN PRODUCING COLUMN

PART I. RECTIFICATION TOWERS

The rectifying column is a device for bringing a current of liquid and a current of vapor into intimate countercurrent contact. Two classes of rectifying columns in general use are the tray and the packed type towers.

In the tray tower intimate contact of liquid and vapor is achieved by bubbling vapor through liquid on a series of trays or plates. The contact is stepwise countercurrent since the liquid flows from tray to tray downward to the stillpot while the vapor rises, bubling through the liquid on each tray, until the summit of the tower is reached. At the top the vapor is condensed and at least partly returned to the plate of the tower as reflux.

In the packed column the trays are replaced by packing. Packings are of many types but all have the primary function of bringing the descending liquid and the ascending vapor into most intimate possible contact. Contact between liquid and vapor in the packed tower is continuous, in contrast to the tray tower where contact takes place only on the trays. The tray column is probably the more widely used of the two since the factors which affect the efficiency of separation are more easily visualized and controlled in this type.

Tray columns have been built and tested for the separation of liquid air which have achieved a lower H.E.T.P.* than has been possible with packed columns under similar conditions[1] (1.9 inches).

Tray columns, however, have disadvantages. The trays must be carefully constructed and installed to prevent large inefficiencies or completely inoperable conditions. The labor of tray construction and installation is relatively great. The amount of liquid hold-up is large, and therefore, the time required to get the unit into operation is relatively long. Also, more care is necessary in shutting down a tray tower since all the trays are filled with liquid which must be evaporated.

Packed towers have many potentialities[2] which for various reasons never seem to be fully realized. Probably the chief cause for the failure of packed towers to work at maximum efficiency is the tendency of the liquid and vapor to find channels in the packing, thereby defeating the purpose of the packing.

---

*H.E.T.P. - An abbreviation for the height equivalent to one theoretical plate. The theoretical plate is the perfect tray; one in which the vapor rising is in perfect equilibrium with liquid on the tray.

[1]Lobo, W.E., and Williams, Brymer, op. cit., Summary, p. 2.

[2]Ibid., p. 118.

Some advantages of packed towers may however be exploited. The higher thruput rate of packed towers allows smaller construction. The small amount of liquid held in the tower and the use of light weight, low heat capacity packings, materially shortens the time required to get a liquid air plant into operation. The main feature of packed towers, however, is simplicity of construction. Packing a tower in the majority of cases means dumping into the shell the desired filling material.

In small diameter columns H.E.T.P. values as low as 1.2 inches have been achieved.[3] However, the effect of increasing H.E.T.P. with increasing diameter of the column is a distinct disadvantage in the use of packings.[4] There is the possibility of overcoming the bad effect of increasing diameter by placing several small columns in parallel. Such a device can be made compact and, with reasonable simplicity.

There are then, three general tower constructions which appear feasible.

(1) The tray tower.
(2) The packed column of appropriate diameter and packing height.
(3) The multiple packed column

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[3]Lobo, W.E., and Williams, Brymer, op. cit., p. 32-A.

[4]Ibid., p. 124, 125.

(1) The tray tower will not be considered here since nothing can be added at this point to designs previously reported.[5] The difficulty of constructing such a tower practically eliminates the possibility of building one in our shops.

(2) The simple packed column offers the easiest construction, but also requires the most head room.

(3) The multiple packed column offers for only slightly increased complexity greater compactness and efficiency. The use of several small packed tubes in parallel allows advantage to be taken of the best efficiency of the packings while at the same time any desired amount of material may be processed.

Approximately the same type and size kettle and condenser are required irrespective of the type of tower chosen.

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[5]Lobo, W.E., and Williams, Brymer, op. cit.

PART II. TOWER CALCULATIONS

As noted in CHAPTER V, it is necessary to operate a nitrogen producing column at higher pressures than atmospheric pressure in order that the bottoms liquid may be used in the condenser at atmospheric pressure. The pressure at which the column should be operated depends upon the temperature difference necessary in the condenser. For the purpose of these calculations the pressure of 75 psia. has been chosen as the operating pressure since, (1)it is approximately the correct pressure and, (2)the equilibrium data available are for this pressure. Commercial columns are known to vary the pressure to suit the conditions.

In addition to the pressure, the following data are necessary to fix the size of the rectification system needed for this plant:

1. Product purity.
2. Number of theoretical plates needed to achieve the desired product.
3. The column traffic.
4. Packing characteristics.
5. Equilibrium and enthalpy data.[6]

These data are discussed briefly below:

(1) Inasmuch as the separation of nitrogen from air is not difficult, a 99.5 mole per cent product will be designed for.

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[6]All thermodynamic and equilibrium data are taken from Lobo, W.E., op. cit.

(2) Under the conditions assumed, four theoretical plates are sufficient to obtain 99.5 mole per cent nitrogen from the air.[7] Also, it can be shown that the feed plate is the stillpot.[7]

(3) The traffic in the column is determined by the amount of vapor which enters in the feed. Figure 10 shows the results of heat and material balance around the column[8] from which the column traffic may be determined.

The feed entering from the liquefier has enthalpy of 25.6 B.T.U. per pound at 500 psia., and upon isenthalpic expansion to 75 psia. the mixture contains 21.5 weight per cent liquid. However, the heat leak into the column has the effect of vaporizing more liquid and therefore must be taken into account. For purposes of calculation the heat leak is assumed to be added into the stillpot and removed in the condenser. Two B.T.U. per pound were assumed for the oxygen stripping column. There is the possibility of a small additional heat leak, so that 2.5 B.T.U. per pound will be used here.

The feed into the stillpot, then, has an enthalpy of 25.6 + 2.5 = 26.1 B.T.U. per pound. This enthalpy at 75 psia. corresponds to a mixture containing 81.7 weight per cent

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[7]See Appendix C for details of theoretical plate calculations.

[8]See Appendix D for heat and material balance.

A3

FIGURE 10 - A diagram of the type rectifying column necessary to produce liquid nitrogen with temperature, pressure, enthalpy and weight per hour of various material streams.

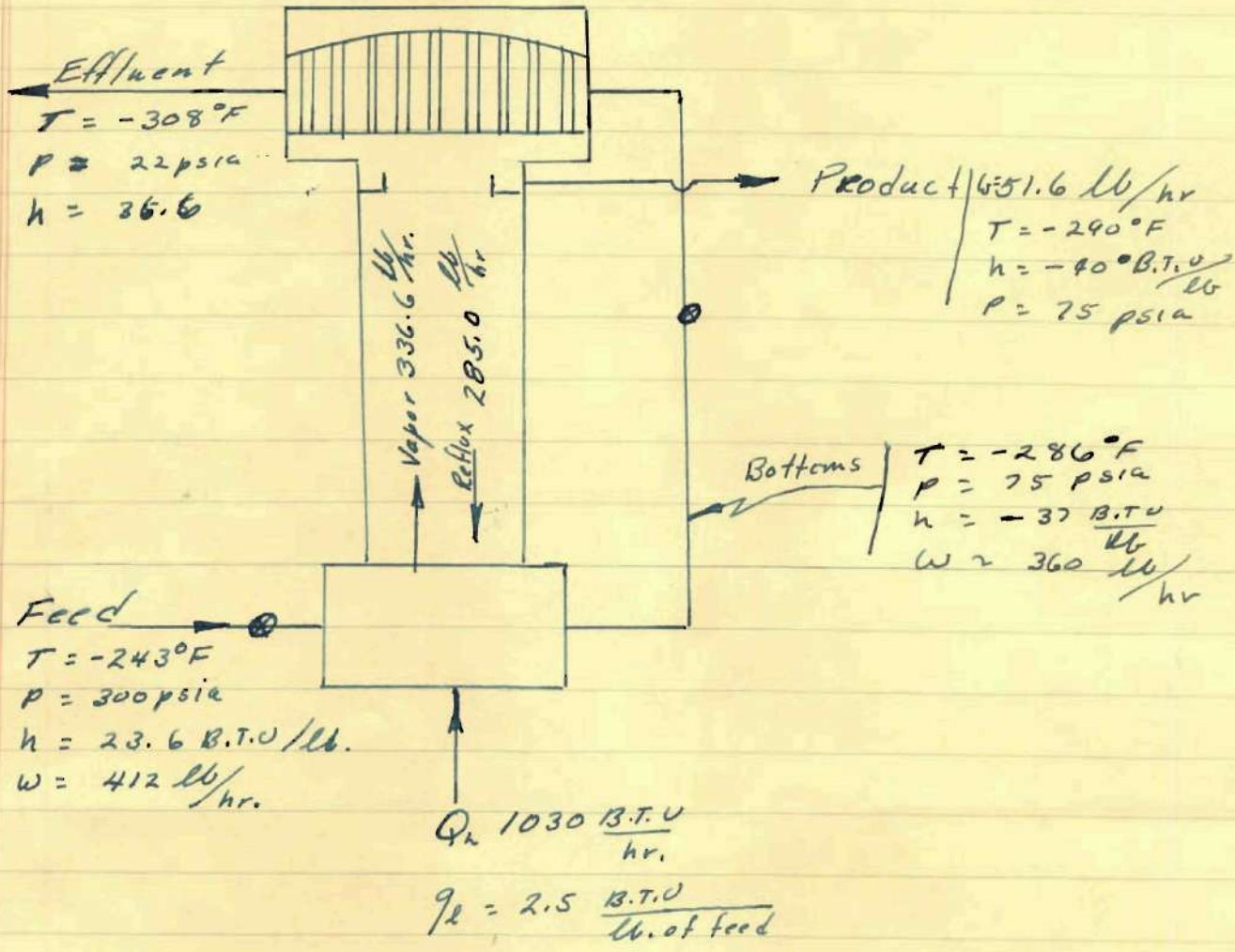
Effluent
T = -308°F
P = 22 psia
h = 36.6
Product
G = 51.6 lb/hr
T = -290°F
h = -40 B.T.U/lb
P = 75 psia
Vapor 336.6 lb/hr.
Reflux 285.0 lb/hr
Bottoms
T = -286°F
P = 75 psia
h = -37 B.T.U/lb
W = 360 lb/hr
Feed
T = -243°F
P = 300 psia
h = 23.6 B.T.U/lb.
W = 412 lb/hr.
Qr 1030 B.T.U/hr.
qr = 2.5 B.T.U/lb. of feed

Figure 10

vapor. Since 412 pounds[9] of material were determined to enter the column, 412 x 0.817, or 336.6 pounds per hour must be vapor.

(4) The allowable traffic for packed columns varies somewhat, but most good packings have flooding points of at leat 1,500 pounds per square foot, per hour.[3] As efficiencies of packings increase with thruput[10] it is desirable to operate at velocities close to flooding. Thirteen hundred pounds per square foot, per hour is chosen as a good average operating value.

Tower Area.--The area of the column required is found by utilizing the thruput (3), and the allowable vapor velocity (4). The area is:

$$\frac{336.6 \text{ lb.}}{\text{hr.}} \times \frac{\text{hr. ft.}^2}{1300 \text{ lb.}} = 0.259 \text{ ft.}^2$$

which is approximately equivalent to a single 7 inch diameter tube. In a multiple column tower four 3.5 inch diameter tubes suffice.

Tower Height.--The tower height is directly dependent upon the H.E.T.P. In general, a given packing will increase its H.E.T.P. with the diameter of the tower for a fixed thruput and system.[11] For a tube 7 inches in diameter the H.E.T.P.

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[9] Chapter VI, p.

[10] Lobo, W.E., and Williams, Brymer, op. cit., p. 52-B.

[11] Robinson, C.S., and Gilliland, E.R., Elements of Fractional Distillation, 3rd. ed., McGraw-Hill, New York, N.Y., 1939, p. 220.

for a good packing is given as approximately 7 inches.[4] In tubes 4 inches and less in diameter the H.E.T.P. is of the order of 2 to 3 inches.[4]

In the single packed column of 7 inch diameter a minimum packed height of 28 inches is indicated. A multiple column, utilizing an H.E.T.P. of 3 inches, would require a packed height of only 12 inches.

All data given here are for $\frac{1}{4}$ inch by $\frac{1}{4}$ inch packing. It is reported to have been found that packing shapes larger than one-eighth the tower diameter tend to throw the liquid to the walls and leave the center dry.[12,13] In systems which operate above room temperature this effect is undoubtably bad since heat leak also tends to condense the liquid on the walls. However, in systems operating below room temperature it would seem that heat leak would tend to dry the walls and drive the liquid to the center of the column. It would be interesting to determine if these two effects would compensate each other in low temperature systems.

The best type of packing to use, while subject to some debate, is indicated by the characteristics which are desired. In this case some desirable features are: low hold-

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[12]Robinson, C.S., and Gilliland, E.R., op. cit., p. 219.

[13]Walker, W.H., Lewis, W.K., McAdams, W.H. and Gilliland, E.R., Principles of Chemical Engineering, 3rd. ed., McGraw Hill, New York, N.Y., 1939, p. 551.

up, low heat capacity, large thruput impervious to liquid air and, of course, low H.E.T.P. Practically all these qualities are found in aluminum Lessing rings.[3] Lessing rings are hollow cylinders of diameter equal to height with a verticle partition up the center. The design will be to use this type packing.[14]

Tower Design.--The multiple column packed tower has so many more interesting possibilities that its design will be considered here in preference to the single packed tower or the tray tower.

Figure 11 is a sketch of a proposed tower design. Four 3.5 inch diameter columns are mounted together on flanged plates. The lower mounting plate serves as a cover for the stillpot. The upper plate functions as a collector for the condensate and also to hold the condenser.

Since, roughly, the same kettle and condenser would be required irrespective of the type of column this system allows any other type column to be substituted for the multiple packed tower provided only that suitable flanges are included.

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[14]Although aluminum Lessing rings are mentioned here, any of a number of packings are equally good. Some of the packings which have proved to be effective are: Berl saddles, ¼ inch glass rings, 5/32 inch brass show eyelets, wire gauze saddles, and Stedman packing. Both aluminum Lessing rings and aluminum Berl saddles have been mentioned in the literature. The lightness and low heat capacity of aluminum would be a definite advantage in a liquid air fractionating system.

Copper tubes for the multiple column tower are desirable to minimize any effect the difference in temperatures on the opposite sides of the columns may have, since the adjacent sides of the tubes will naturally tend to be colder than the opposite sides.

The wall thickness necessary is given by:

$$t = \frac{\text{pressure x mean radius}}{\text{allowable stress}}$$ [15]

The allowable stress for copper varies considerably from annealed to the hard-drawn material. While it seems perfectly feasible to use hard-drawn tubes the additional wall thickness indicated by the lower allowable stress of annealed copper is not excessive. The allowable stress is chosen as 70 per cent of the yield point of annealed copper as given by Seely.[16] Based on the ultimate strength of copper this gives a factor of safety of 14.

$$t = \frac{75 \text{ psi. x } 3.5 \text{ in.}}{.7 \text{ x } 3200 \text{ psi.}} = .117 \text{ inches}$$

A tube with a 1/8 inch wall is sufficiently strong.

In a similar fashion the thickness of a spun copper

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[15]Roark, R.J., Formulas for Stress and Strain, McGraw-Hill, New York, N.Y., 1938, p. 226.

[16]Seely, F.B., Resistance of Materials, 2nd ed., John Wiley and Sons, New York, N.Y., p. 421

kettle with dished head can be shown to require a wall thickness of .312 inches.

The kettle has an approximate capacity of 25 liters when liquid is six inches from the top. Three openings are required in the kettle: a feed entrance, an outlet for liquid to the condenser, and a small drain line in the bottom.

The condenser is a shell and tube type, having 500 $\frac{1}{4}$ inch outside diameter, six inch long copper tubes. The surface area is approximately 16.3 square feet.

The nitrogen vapor rising from the columns is deflected and spread out to provide reasonably uniform distribution over the condenser face area. The vapor enters the open ended tubes from below and travels upward until it is condensed on the tube wall. The liquid nitrogen runs down the wall in a liquid film.

Condenser Calculations.--The conditions under which the logarithmic mean overall temperature difference is correct average temperature difference[17] are closely approached in this condenser. The cooling liquid from the kettle is throttled from 75 psia. into the condenser at 18 psia; the temperature falls to -312°F. The liquid coolant is vaporized and the vapor leaving the condenser is approximately at saturation at -308°F. The nitrogen enters the condenser as

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[17]McAdams, William H., Heat Transmission, 2nd. ed., McGraw-Hill, New York, N.Y., 1942, p. 141.

a saturated vapor and is assumed to leave as a saturated liquid at the same temperature of -290°F.

The log mean temperature difference then is:

$$tm = \frac{(312 - 290)^{\circ}F. - (300 - 290)^{\circ}F.}{\ln \frac{(312 - 290)}{(308 - 290)}}$$

$$= 20^{\circ}F.$$

Actual commercial columns are known to operate at temperature differences of 14°F. in the condenser.

The amount of heat to be transfered in the condenser is 25,950 B.T.U. per hour[18] and is equal to the overall coefficient of transfer, U, multiplied by the area of transfer, A, and the temperature difference, tm.

$$25950 = UA\ tm$$

A perusal of reported values for U under various conditions[19,20] indicates that a value of U equal to 200 is probably conservative.

Solving for the area of transfer:

$$25950 = 200\ A \times 20$$
$$A = 6.48\ ft.^2$$

The 16 square feet provided should be ample.

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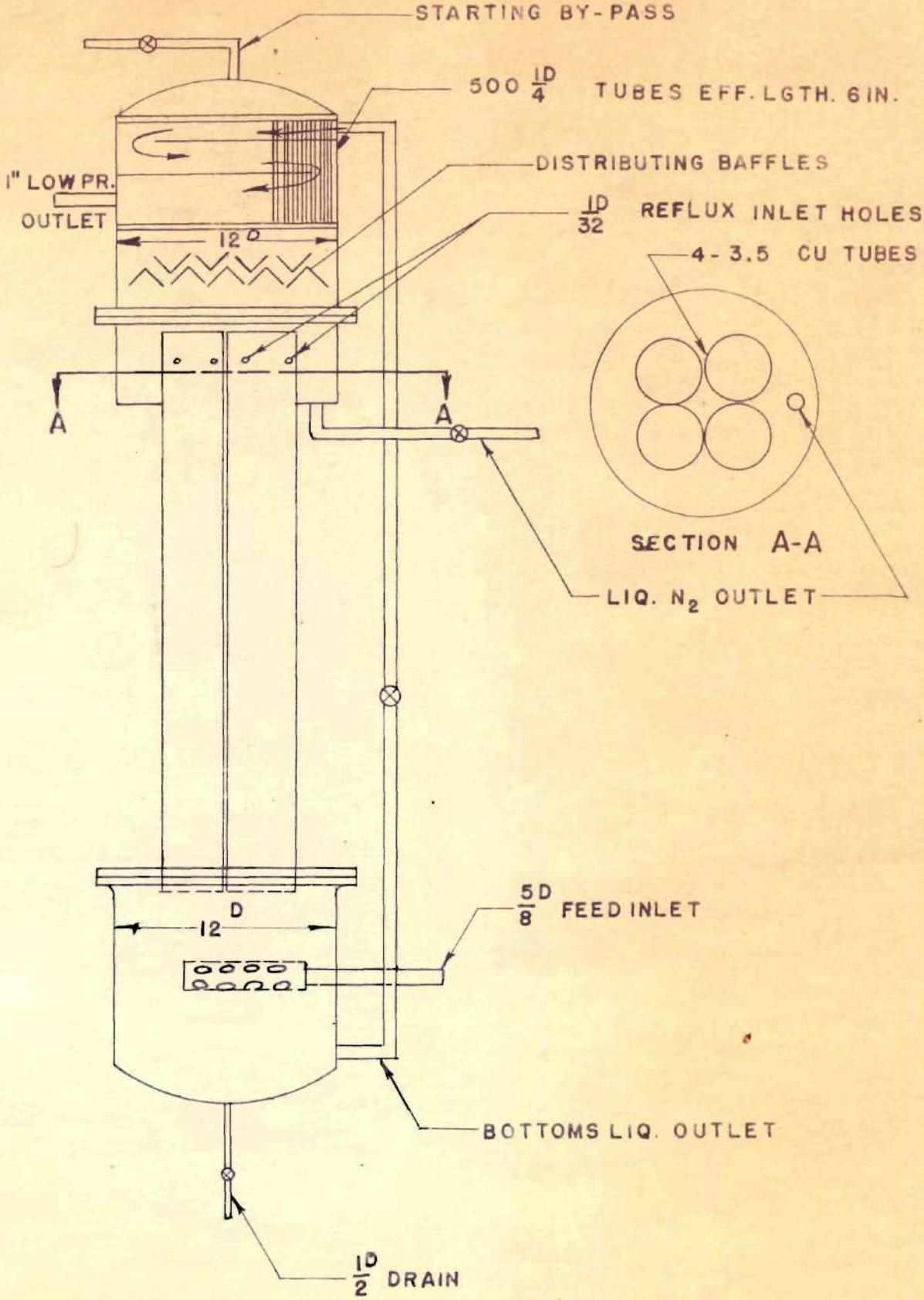
[18] See Appendix D for detailed heat balance.

[19] McAdams, W.H., op. cit., p. 271.

[20] Robinson, C.S., and Gilliland, E.R., op. cit., p. 230

FIGURE 11 - Proposed multicolumn rectifying tower for liquid nitrogen production.


STARTING BY-PASS
500 1D/4 TUBES EFF. LGTH. 6 IN.
DISTRIBUTING BAFFLES
1" LOW PR. OUTLET
1D/32 REFLUX INLET HOLES
12 D
4 - 3.5 CU TUBES
A
A
SECTION A-A
LIQ. N2 OUTLET
5D/8 FEED INLET
12 D
BOTTOMS LIQ. OUTLET
1D/2 DRAIN

Distribution and Drawoff of Liquid Nitrogen.--To a certain extent the reflux ratio is controlled by the amount of nitrogen withdrawn. In order to prevent each withdrawal from upsetting the reflux to the column it is necessary to arrange to have the reflux enter the column at some point below the top edge where any withdrawal would have the effect of momentarily stopping the reflux. The nitrogen return to the column is accomplished through the small holes shown in the sides of the tube walls just below the top. As long as the liquid level is above these holes reflux will be maintained. Since the rate of draw off is small relative to the total amount of liquid condensed, (approximately 1/7) trouble is not anticipated at this point.

CHAPTER VIII

TEST RUNS OF LIQUID AIR PLANT

Two test runs of the plant as a liquid air producing plant have been made. To change the plant from a liquid oxygen plant to a liquid air plant a by-pass, with appropriate valves, was placed around the column. Thus, the feed from the liquefier was introduced into the stillpot rather than at the top of the rectifying section and therefore liquid of composition closely akin to air was collected. This change was made to obviate the dangers of handling liquid oxygen.

The first run was started on April 27, 1948, at 11:45 A. M.[1] The late start was due to difficulties experienced in starting the expansion engine. The valve settings on this machine were not correct and some adjustment was necessary to get it to run. Once started, however, it ran reasonably well.

This first test can only be termed exploratory. It was the first time any of those present had ever seen a liquid air plant operate, to say nothing of operating one. The operation was carried on from the Operating Manual in true cook-book fashion. The sparseness of the data arises from the fact that the operators were too busy to make recordings.

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[1] See Appendix E for details of run.

The temperatures fell rapidly during the first two hours of operation but after that they began to level off and further decreases were extremely slow in coming. At the end of six hours no liquid was present in the kettle and the operation was discontinued even though the temperatures were still decreasing. It is believed now that liquid could have been made if the proper operating proceedure had been followed.

However, several changes were indicated by this operation. At several places on the outside of the interchanger large "sweat spots" developed, indicating a sizable heat leak at those points. The solenoid valve controlling the air intake to the expansion engine failed to work satisfactorily in that it frequently closed, thereby stopping the engine. The failure of the expansion engine exhaust to get as cold ($-175^{\circ}C.$) as the Operating Manual stated it should led to the belief that the valves of the engine were not properly set. This was later checked and found to be not true but other adjustments were necessary.

The second run was made with much more confidence than the first and the production of liquid air justified this confidence. The fact that a reasonably complete log[2] of the operation was kept is indicative of the calmer state of affairs existing during this run.

The second test was made on May 4, 1948, one week

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[2] See Appendix E.

after the first attempt. During that week the changes indicated by the first operation were made. The expansion engine valves were checked for timing and clearance, additional insulation was added to the interchanger and the solenoid valve was repaired. Two additional thermocouples were added before the second test; one thermocouple was placed on the liquefier air intake line and the other on the stillpot feed line just after the throttle valve. The additional temperature information was of considerable aid in the operation of the throttling process. It was possible to ascertain that liquid was being produced even though the liquid level gage on the kettle failed to indicate satisfactorily.

Liquid was first observed in the kettle about six hours after starting. The Operating Manual states that from five to six hours are necessary to get the plant into production. No liquid was withdrawn for another hour and a half; after which time the liquid air was transferred steadily until exhausted. The transfer required one hour and twenty-five minutes. When all the liquid was exhausted from the kettle the plant was shut down.

An attempt was made to collect the liquid air in 25 liter thermos cans and some 45 pounds were collected, but it proved to be a very wasteful process. The neck of the can is small and returning gas blew large amounts of liquid out. Probably only fifty per cent of the liquid transferred was

collected.

The estimated production of 90 pounds for the three hour period is somewhat below the rated average production of 50 pounds per hour but it can definitely be stated that equilibrium conditions were not achieved and production was probably not at its peak level.

The temperature drop in the expansion engine was still not as great as desired. Some additional adjusting seems to be necessary. The compressor showed signs of leaks around several exhaust manifolds and since no more than enough air was delivered to the interchanger this defect should be repaired. The solenoid value worked well until a stroke of lightning caused a power failure on the 110 volt A.C. circuit. (The 220 volt compressor motor circuit was not affected). Thereafter the valve failed to function properly. It will probably have to be replaced.

The overall results of the operation of this plant have been encouraging and with proper attention to care and maintenance of the machinery a satisfactory end result should be obtained.

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APPENDIX A

OXYGEN LOST TO EFFLUENT GAS

The percentage of oxygen lost to the effluent gas from the column is necessary for the calculation of the material balance in CHAPTER VI. In the following section the reasoning used to arrive at the assumed 10 per cent loss will be set forth.

Ordinarily the loss of oxygen can best be estimated by considering the conditions of the feed plate. However, the exact feed plate conditions are not known and it is necessary to make some assumption about the condition of the feed at that point. The feed condition is fixed by its temperature as it emerges from the condenser coil. The condenser coil is immersed in liquid oxygen boiling at the column pressure of 22 psia. (-291°F.). The length of the coil is not known but the inside diameter is .041 feet.

Reynolds number is $\frac{DG}{u}$

where D = diameter in feet

G = mass flow rate in $\frac{\text{lb.}}{\text{sec. ft.}^2}$

u = viscosity in $\frac{\text{lb.}}{\text{ft. sec.}}$

G, the mass flow rate is estimated to be approximately 7.3 pounds per second. The viscosity is of the order of 0.10 centipoise. Reynolds number for these conditions is therefore large (about 2.5×10^6).

The large Reynolds number, copper tube and the fact that the heat transfers from a condensing vapor to a boiling liquid, both of low viscosity, all point to excellent conditions for the transfer of heat.

A three degree temperature difference in the reboiler will be assumed. The temperature and enthalpy of the liquid just prior to the throttle valve are then -288°F. and -38.5 B.T.U. per pound, respectively. Upon isenthalpic expansion to 22 psia. the temperature falls to -312°F.

The following analysis of the throttling process shows that 14.5 weight per cent of the liquid flashes into vapor.

Referring to Figure 6, the air enters the condenser coil at -243°F. and 300 psia. The air is cooled at constant pressure to -288°F. where the enthalpy is -38.5 B.T.U. per pound isenthalpic expansion takes place at the throttle valve.

The per cent liquid may be determined by the following relation:

$$\frac{h_{\text{sat. vap.}} - h_{\text{product}}}{h_{\text{sat. vap.}} - h_{\text{sat. liq.}}} \times 100$$

$h_{\text{sat. vap.}}$ at 300 psia. = 35.5 B.T.U./lb.

$h_{\text{sat. liq.}}$ at 300 psia. = -51.0

h_{product} at 300 psia. = -38.5

then the per cent liquid is found to be 85.5 weight per cent and the vapor 14.5 weight per cent.

This is then, the condition at the point of feed: 14.5 per cent vapor, 84.5 per cent liquid. The composition of the liquid and vapor is not readily discernable.

The theoretical minimum amount of oxygen is lost if the vapor leaving the column has the composition (6.4 per cent oxygen) of vapor in equilibrium with liquid air. This excepts the improbable case of a sub-cooled liquid feed. Any flashing occuring upon throttling tends to increase the oxygen lost. The composition of the flashed vapor is higher in nitrogen than the liquid since nitrogen is the more volatile component. The liquid left is therefore richer in oxygen than air and the vapor in equilibrium with this liquid is richer in oxygen than the vapor over liquid air. The maximum oxygen loss, under equilibirum conditions, would occur if the flashed vapor were pure nitrogen. For this condition, and 14.5 weight per cent flash, the vapor in equilibrium with the liquid residue of the flashing would contain 7.5 mole per cent oxygen. For equilibrium conditions then, the oxygen loss is bracketed between 6.4 and 7.5 mole per cent.

In columns of this type, operating under varying conditions, the values for oxygen loss in the effluent gas vary from as low as 7.3 to as high as 13.7[1] mole per cent. Simple stripping towers such as this are not used commercially and

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[1]Lobo, W. E., and Williams, Brymer, op. cit., p. 161

little data are available, however, one writer[2] assumes 9 mole per cent easily attainable, but inasmuch as this system has not proved a particularly stable one in operation, 10 mole per cent oxygen loss will be assumed.

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[2]Ruhemann, M., op. cit., p. 139

APPENDIX B

ENTHALPY BALANCE ON LIQUID OXYGEN PRODUCING PLANT

The procedure in these calculations will be to make the assumptions necessary to complete an enthalpy balance on the distillation column and then make another balance on the rest of the plant up to the column and compare the results of the two at that point.

The Column.--The heat into the column is comprized of two parts: (1) Heat brought in by the feed, (2) Heat leak. The heat in must be equal to the heat out in the effluent gas and the liquid oxygen product. Thus,

$$H_{feed} + H_{leak} = H_{eff.} + H_{product}$$

If a basis of 1 hours operation is chosen,

$$H_{product} = \frac{50 \text{ lb. oxygen}}{\text{hr.}} \times \text{enthalpy } (-56.5 \frac{\text{B.T.U.}}{\text{lb.}})$$

$$= -2825 \frac{\text{B.T.U.}}{\text{hr.}}$$

$$H_{eff.} = (412-50)\text{lb. effluent} \times 37 \frac{\text{B.T.U.}}{\text{lb.}}$$

$$= 13{,}394 \frac{\text{B.T.U.}}{\text{hr.}}$$

Dodge[1] has assumed the heat leak to be 90 B.T.U. per pound mole of air entering an air plant. Others[2] have found

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[1] Dodge, B.F., Chemical Engineering Thermodynamics, McGraw-Hill, New York, N.Y., 1944, p. 479.

[2] Lobo, W.E., and Williams, Brymer, op. cit., p. 17.

in plants of approximately the same size and smaller that the heat leak into the column was 2 B.T.U. per pound. Since the latter figure represents results on systems not very different than the one under consideration it will be used and will be assumed to be 20 per cent of the total heat leak.

For the column then,

$$H_l = \frac{412 \text{ lb. air}}{\text{hr.}} \times \frac{2 \text{ B.T.U.}}{\text{lb.}} = 824 \frac{\text{B.T.U.}}{\text{hr.}}$$

$$H_{feed} + 824 = 13{,}394 + (-2825)$$

$$H_{feed} = 9{,}745 \frac{\text{B.T.U.}}{\text{hr.}}$$

the enthalpy of the feed, $h_f = \frac{9{,}745 \text{ B.T.U.}}{412 \text{ lb.}} = 23.6 \frac{\text{B.T.U.}}{\text{lb.}}$

At this point an enthalpy balance will be made around the entire plant excepting the column.

The heat load consists of the following:

(1) Condensation of water vapor.
(2) Freezing of water vapor.
(3) Condensation of carbon dioxide.
(4) Heat Leak.
(5) Cooling of all air through countercurrent coil.
(6) Cooling air in the liquefier.
(7) Condensation of air in the liquefier.

The refrigeration must be equal to the heat load and is derived from two sources. The main source of refrigeration is the expansion engine exhaust; the other source of cooling is the waste gas from the top of the column.

The heat load will be examined first.

(1) Condensation of water vapor.--The air enters the interchanger saturated with water at 300 psia. and 90°F. The compressed volume of this air is,

$$\frac{1123 \text{ lb. air}}{\text{hr.}} \times \frac{1 \text{ ft.}^3}{1.52 \text{ lb.}} \text{ (3)} = 740 \frac{\text{ft.}^3}{\text{hr.}}$$

From the steam tables[4] the specific volume of saturated water vapor at 90°F. is found to be 468.5 ft.^{3} per pound. At the relatively low pressure of 300 psia., total pressure probably has little effect on the vapor pressure of water.[5] Therefore,

$$\frac{740 \text{ ft.}^3}{\text{hr.}} \times \frac{1 \text{ lb. water}}{468.5 \text{ ft.}^3} = 1.58 \frac{\text{lb. water}}{\text{hr.}}$$

passes into the warm leg as vapor.

The air leaves the warm leg at about 41°F.[6] and 300 psia. at which temperature the specific volume water vapor is 2365 ft.^{3} per pound.[4]

The air volume at 41°F. is,

$$\frac{1123 \text{ lb. air}}{\text{hr.}} \times \frac{1 \text{ ft.}^3}{1.68 \text{ lb. air}} \text{ (3)} = 670 \frac{\text{ft.}^3}{\text{hr.}}$$

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[3] Lobo, W.E., op. cit., Curve No. 815.60.

[4] Lange, A.L., Editor, Handbook of Chemistry, Handbook Publishers, Inc., Sandusky, Ohio, 1937, p. 1220.

[5] Perry, J.H., op. cit., p. 392.

[6] Operating Manual, p. VIII-4.

thus,

$$\frac{670 \text{ ft.}^3}{\text{hr.}} \times \frac{1 \text{ lb. water}}{2365 \text{ ft.}^3} = .28 \text{ lb. water}$$

entering the cold leg at 300 psia. and 41°F.,

$$1.58 \text{ lb.} - 1.28 \text{ lb.} = 1.3 \text{ lb. of water}$$

condensed in the warm leg,

the enthalpy of vapor at 90°F. = 1079.0 $\frac{\text{B.T.U.}}{\text{lb.}}$

the enthalpy of liquid at 41°R = 8.0

the change in enthalpy = 1071.0 $\frac{\text{B.T.U.}}{\text{lb.}}$

$\frac{1.3 \text{ lb.}}{\text{hr.}}$ of water condensed x h = 1391 $\frac{\text{B.T.U.}}{\text{hr.}}$

$$H_1 = 1391 \frac{\text{B.T.U.}}{\text{hr.}}$$

All the condensed water is removed from the warm leg in traps.

(2) Freezing of water vapor.--In the cold leg 0.3 pounds per hour of water is condensed and frozen. This liberates an insignificant 365 B.T.U. per hour, in the cold leg.

$$H_2 = 365 \frac{\text{B.T.U.}}{\text{hr.}}$$

(3) Condensation of carbon dioxide.--The normal amount of carbon dioxide in clean air is .03 mole per cent, but larger values are the rule in populated areas.[7]

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[7] Quinn, E.L. and Jones, C.L., Carbon Dioxide, A.C.S., Monograph 72, New York, N.Y., Reinhold Pub. Corp., 1936, p. 19.

.07 mole per cent seems to be a satisfactory value.

$$\frac{.07 \text{ moles } CO_2}{100 \text{ moles air}} \times \frac{1123 \text{ lb. air/hr.}}{29 \text{ lb. air/mole}} = 0.0271 \frac{\text{mole } CO_2}{\text{hr.}}$$

Air entering the liquefier is about -160°F. and has a volume of

$$\frac{1123 \text{ lb. air}}{\text{hr.}} \times \frac{1 \text{ ft.}^3}{2.05 \text{ lb.}} = 549 \frac{\text{ft.}^3}{\text{hr.}}$$

The vapor pressure of CO_2 at this temperature is 76 mm Hg.[8] The volume of CO_2 is,

$$\frac{0.0271 \text{ mole } CO_2}{\text{hr.}} \times \frac{300^\circ R}{460^\circ R} \times \frac{760 \text{ mm}}{76 \text{ mm}} \times \frac{359 \text{ ft.}^3}{\text{mole}} = \frac{63.5 \text{ ft.}^3}{\text{hr.}}$$

Up to entering the liquefier no carbon dioxide will have condensed.

At the temperature of air leaving the liquefier, (-245°F.), the vapor pressure of carbon dioxide is practically nil, so that nearly all will condense in the liquefier.

The heat of sublimation of carbon dioxide is given as 252 B.T.U. per pound.[9]

412 pounds of air go to the liquefier, therefore,

$$\frac{412 \text{ lb. air}}{29 \text{ lb. air/mole}} \times \frac{.07 \text{ mole } CO_2}{100 \text{ mole air}} \times \frac{44 \text{ lb. } CO_2}{\text{mole } CO_2} \times \frac{252 \text{ B.T.U.}}{\text{lb.}} =$$

$$1105 \frac{\text{B.T.U.}}{\text{hr.}}$$

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[8] Quinn, E.L., and Jones, C.L., Carbon Dioxide, A.C.S., Monograph 72, New York, N.Y., Reinhold Pub. Corp., 1936, p. 63

[9] Ibid., p. 70

(4) Heat Leak.--The heat leak of the plant was previously assumed to be four times that of the column. The column heat leak was 824 B.T.U. per hour.

$$H_4 = 4 \times 824 = 3296 \frac{\text{B.T.U.}}{\text{hr.}}$$

(5) Cooling of all air through countercurrent coil.--The entering air is at 90°F and 300 psia.

enthalpy of entering air = 130 $\frac{\text{B.T.U.}}{\text{lb.}}$

enthalpy of air leaving countercurrent coil at -160°F. + 300 psia. = 66

enthalpy difference = 64 $\frac{\text{B.T.U.}}{\text{lb.}}$

$$\frac{1123 \text{ lb. air}}{\text{hr.}} \times \frac{64 \text{ B.T.U.}}{\text{lb.}} = 71{,}800 \frac{\text{B.T.U.}}{\text{hr.}}$$

$$H_5 = 71{,}800 \frac{\text{B.T.U.}}{\text{hr.}}$$

(6) Cooling air in the liquefier.--Only the 412 pounds of air going through the liquefier require further cooling. To remove the sensible heat of the air in the liquefier,

enthalpy, $h_{\text{air in}}$ = 66 $\frac{\text{B.T.U.}}{\text{lb.}}$ at -160°F. & 300 psia

enthalpy of air at dew point = 40

Change in enthalpy = 26 $\frac{\text{B.T.U.}}{\text{lb.}}$

$$\frac{412 \text{ lb.}}{\text{hr.}} \times \frac{26 \text{ B.T.U.}}{\text{lb.}} = 10710 \frac{\text{B.T.U.}}{\text{hr.}}$$

$$H_6 = 10{,}710 \frac{\text{B.T.U.}}{\text{hr.}}$$

$$H_1 + H_2 + H_3 + H_4 + H_5 + H_6 + H_7 = 87{,}672 \frac{\text{B.T.U.}}{\text{hr.}} + H_7$$

The refrigeration has two sources:

(A) Waste gas from the column -

the enthalpy of waste = 37 $\frac{\text{B.T.U.}}{\text{lb.}}$

The effluent gas leaves the last heat exchanger at about 80°F., that is 10°F. colder than the air entering the plant.

the enthalpy of gas at 80°F. = 131.5 B.T.U.

H_A = (412-50) lb. of effluent x (131.5 - 37) = 34300 $\frac{\text{B.T.U.}}{\text{hr.}}$

(B) The expansion engine exhaust. Seven hundred and eleven pounds of air per hour pass through the expansion engine. The air normally enters the engine at about -148°F. and leaves at -270°F.[10] and 19 psia. The enthalpy of the gas leaving the engine is 45.0 B.T.U. per pound.

the enthalpy of the exhaust at 80°F. = 130.0

H_B = 711 lb. x (130.0 - 45.0) = 60,500 $\frac{\text{B.T.U.}}{\text{hr.}}$

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[10] The Operating Manual states the temperature of the exhaust to be about -283°F. Tests and data from logs, however, indicate -270°F. to be more nearly the correct average.

$$H_A + H_B = 94{,}800 \frac{\text{B.T.U.}}{\text{hr.}}$$

Since the enthalpy balance must equal zero,

$$87{,}672 + H_7 = 94{,}800$$

$$H_7 = 7{,}128 \frac{\text{B.T.U.}}{\text{hr.}}$$

7,128 B.T.U. per hour are available for liquefying 412 pounds of air or:- $\frac{7{,}128}{412} = 17.3 \frac{\text{B.T.U.}}{\text{lb.}}$

The enthalpy of saturated air vapor at 300 psia. is,

$$40 \frac{\text{B.T.U.}}{\text{lb.}}$$

Therefore, the enthalpy of the liquid vapor mixture leaving the liquefier is,

$$40 - 17.3 = 22.7 \frac{\text{B.T.U.}}{\text{lb.}}$$

This figure compares favorably with the 23.6 B.T.U. per pound obtained by the heat balance around the column.

APPENDIX C

ENTHALPY BALANCE ON LIQUID NITROGEN PRODUCING TOWER

To make an enthalpy balance for the proposed liquid nitrogen producing tower some assumptions about the terminal operating conditions must be made. Probably the simplest and most logical assumption is that the feed to the tower be exactly the same as the feed to the oxygen producing tower. In effect, for a given operating pressure in the tower, fixing the feed enthalpy fixes all other quanities in the tower excepting the heat leak.

If the weight, enthalpy, and pressure of the air feed to the column is to be fixed, a fixed amount of refrigeration must be supplied to the heat exchangers which means that the enthalpy of the effluent from the condenser must have a fixed value.

The above conditions will be assumed in the following calculations.

Basis. (1) That the heat balance around this column be the same as for the oxygen column except for an additional heat leak of 0.5 B.T.U. per pound of feed.

The original tower effluent furnished 34,300 B.T.U. per hour to cool incoming gas.*

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*Appendix B.

Then,

$$\text{Wt. of eff.} \times \text{h eff.} = 34{,}300 \ \frac{\text{B.T.U.}}{\text{hr.}}$$

The weight of the effluent is the feed less the amount of product drawn off = (412 - D) pounds. The enthalpy change of the effluent depends somewhat on the composition which in turn depends on the product drawn off. Assume as a first approximation that 50 pounds of liquid nitrogen are removed. Then the composition of the effluent is 76.2 mole per cent nitrogen and 23.8 mole per cent oxygen. The effect of this composition change on the enthalpy is less than $\pm\frac{1}{2}$ B.T.U. per pound and is beyond the scope of these calsulations. The enthalpy of the effluent leaving the plant at 80°F. is,

$$131 \ \frac{\text{B.T.U.}}{\text{lb.}}$$

Therefore effluent enthalpy change is (131 - h eff.)
So that

$$(412 - D)(131 - \text{h eff.}) = 34{,}300 \ \frac{\text{B.T.U.}}{\text{hr.}} \qquad (1)$$

Now since the heat balance around the column is the same, the entering feed has an enthalpy of 23.6 B.T.U. per pound and

$$H_{feed} + H_{leak} = H_{product} + H_{effluent}$$

where H represents $\frac{\text{lb.}}{\text{hr.}} \times \frac{\text{B.T.U.}}{\text{lb.}} = \frac{\text{B.T.U.}}{\text{hr.}}$

$$H_{feed} = \frac{412 \text{ lb. feed}}{\text{hr.}} \times 23.6 \frac{\text{B.T.U.}}{\text{lb.}} = 9730 \frac{\text{B.T.U.}}{\text{hr.}}$$

$$H_{leak} = \frac{412 \text{ lb. feed}}{\text{hr.}} \times \frac{2.5 \text{ B.T.U. heat leak}}{\text{lb. of feed}} =$$

$$1030 \frac{\text{B.T.U.}}{\text{hr.}}$$

$H_{product}$ = D lbs. nitrogen x enthalpy of saturated liquid nitrogen at 75 psia. = D (-40) and

$$H_{effluent} = (412 - D)\frac{\text{lbs.}}{\text{hr.}} \times h_{eff.}$$

then substituting

$$10760 = (412 - D)h_{eff.} - 40 D \qquad (2)$$

$$(412 - D)(131 - h_{eff.}) = 34{,}300 \qquad (1)$$

Solving for D

$$54{,}000 - 131 D - 10760 - 40 D = 34{,}300$$

$$D = 51.6$$

$$h_{eff.} = \frac{10760 + 40 D}{(412 - D)} = 36.6 \frac{\text{B.T.U.}}{\text{lb.}}$$

The Composition of the bottoms.--Composition of original feed is $\frac{412 \text{ lb. air}}{29 \text{ lb./mole a}} \times .79 = 11.22$ moles nitrogen in feed

$\times .21 = 2.98$ moles oxygen in feed

14.20 moles air feed per hour.

51.6 pounds of nitrogen product leave the tower per hour.

$$\frac{51.6 \text{ lbs.}}{28 \text{ lbs./mole}} = 1.84 \text{ moles nitrogen product per hour.}$$

14.2 moles feed - 1.84 moles nitrogen product leaves 12.36 moles of bottoms liquid.

The moles of nitrogen in bottoms is moles of nitrogen in the feed - moles of product

11.22 - 1.84 = 9.38 moles nitrogen in the bottoms

Composition of bottoms

$\frac{9.38}{12.36}$ = 76 mole per cent nitrogen

And (by difference) 24 mole per cent oxygen

APPENDIX D

CALCULATION OF THE NUMBER OF THEORETICAL PLATES

The method used here to determine the number of theoretical plates required to obtain 99.5% nitrogen is the McCabe-Thiele method. This method is explained in any standard textbook on distillation. The method of Ponchon and Savarit[3] was also used and gave a virtually identical answer but since the simpler McCabe-Thiele diagram gives the same result it will be used to illustrate the calculation.

A plot of the equilibrium data[1] for nitrogen at 75 psia. was made. The slope of the operating line was determined from the calculated condenser duty and product withdrawal rate.

25,950 $\frac{\text{B.T.U.}}{\text{hr.}}$ are transferred in the condenser.[2]

51.6 lbs. of product are withdrawn per hour.

The enthalpy change on condensation is 75 $\frac{\text{B.T.U.}}{\text{lb.}}$

Since the slope of the operating line is given by the ratio, $\frac{\text{moles of liquid flowing down the column}}{\text{moles of vapor rising}}$

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[1] Lobo, W.E., op. cit., Curve No. 815.52.

[2] See Appendix D for heat balance.

[3] Dodge, B.F., op. cit., p. 640.

At the top of the column this ratio is given by,

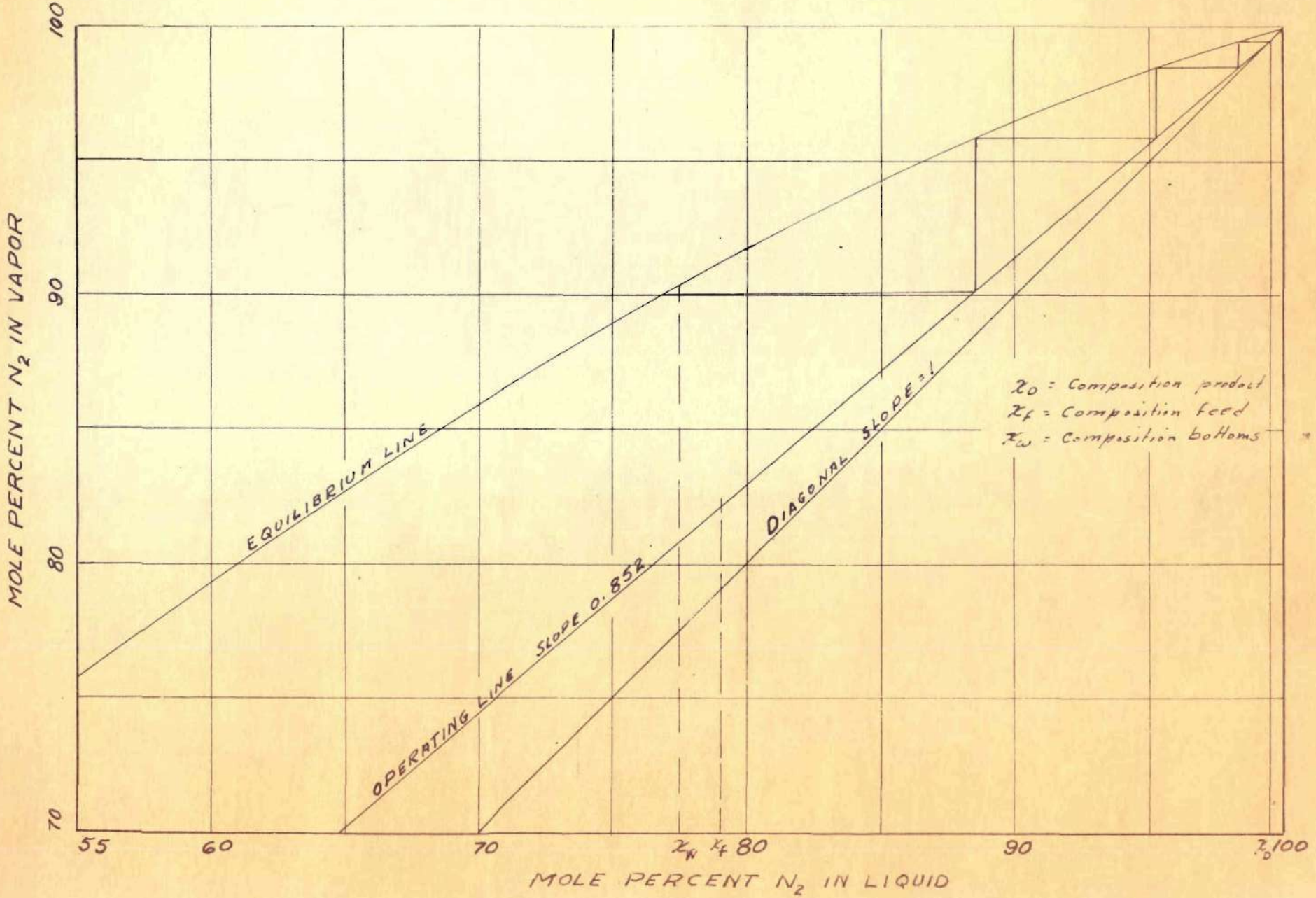
$$\frac{\frac{25950 - 51.6\,(75)}{75}}{\frac{25950}{75}} = .852$$

this is the slope of the operating line.

A line of this slope was drawn and the steps made on the diagram as indicated.

The bottoms composition line x_w is crossed on the fourth plate.

Since the composition of the feed as well as that of the bottoms were reached on the same plate it is obvious that the condition of the feed is of no consequence and that feeding into the stillpot is indicated.


MOLE PERCENT N_2 IN VAPOR
100
90
80
70
55
60
70
x_w
x_f 80
90
x_D 100
MOLE PERCENT N_2 IN LIQUID
EQUILIBRIUM LINE
OPERATING LINE SLOPE 0.852
DIAGONAL SLOPE = 1
x_D = Composition product
x_f = Composition feed
x_w = Composition bottoms

APPENDIX E

DATA OF TEST RUNS OF LIQUID AIR PRODUCING PLANT

April 27, 1948

COMPRESSOR PRESSURE STAGES				INTERCHANGER PRESSURES						TEMPERATURES °C.							
	Air In Cold Leg	Air Out Cold Leg		Exp. Eng. Inlet	Air Out Liq.	Filter	Column			T.C. 1	T.C. 2	T.C. 3	T.C. 4	T.C. 6	Exp. Eng. Speed R.P.M.	Kettle Liq. Level In.	
Time	1st	2nd	3rd	#52	#53	#54	#55	#57	#58								REMARKS
1:00	18	84	252	252	252	250	251	0	0	20	-63	-45	-120		350	0	Start 11:45 A.M.
1:30	19	85	245	252	253	250	252	0	0	18	-59	-31	-122		400	0	Trouble with solenoid valve
1:55	19	85	275	270	270	265	270	0	0	18	-59	-45	-140		425	0	
2:25				250	255	250	255	0	0	18	-60	-56	-147		425	0	
2:55	19	83	240	250	250	245	250	0	0	15	-64	-67	-152		460	0	
3:45	19	87	265	270	270	260	270	10	0	12	-59	-76	-146	-12	490	0	
4:40	19	87	260	270	270	260	270	22	8	12	-59	-76	-146	-12			
5:30	19	88	280	280	280	275	280	10	7	12	-45	-89	-150	-9	500	0	Shut down 6:00 P.M.

May 4, 1948

Time	COMPRESSOR PRESSURE STAGES 1st	2nd	3rd	INTERCHANGER PRESSURES Air In Cold Leg #52	Air Out Cold Leg #53	Exp. Eng. Inlet #54	Air Out Liq. #55	Filter #57	Column #58	TEMPERATURES °C. T.C. 1 Air Out Warm Leg	T.C. 2 Air Out Cold Leg	T.C. 3 Exp. Eng. Inlet	T.C. 4 Exp. Eng. Exhaust	T.C. 6 Between Sections Cold Leg	T.C. A After Expansion Valve	T.C. B Into Liquefier	Exp. Eng. Speed R.P.M.	Kettle Liq. Level In.	REMARKS
9:25	18	83	250	260	260	255	260	0	0	23	-10	+21	-77	+20			490	0	Start 9:05 using all "B" exchangers except liquefier.
10:15	19	84	270	250	250	245	250	0	0	18	-56	-28	-112	+3			500	0	Start closing valve #33.
10:45	19	88	270	290	290	275	290	0	0	18	-48	-39	-128	-1			490	0	Temperatures of compressor discharge: 1st 325°F;
11:15	19	88	280	280	280	275	280	0	0	18	-50	-50	-138	-5			465	0	2nd 350°F., 3rd 330°F.
11:30	20	88	270	292	292	285	290	0	0	15	-66	-47	-139	-8			500	0	Cooling water 78°F. in; 90°F. out; cooling water
12:00	19	90	270	280	280	270	280	0	0	12	-50	-81	-152	-10	+17	-80	480	0	pressure 55 psi. at compressor. Open valve 12A.
12:30	20	90	280	290	295	285	295	0	4.5	16	-46	-90	-150	-4	-48	-89	480	0	All air intakes about 85-90° F. Liquid air in lique-
1:00	19	89	280	285	290	280	290	0	7.0	14	-48	-100			-62	-118	480	0	fier. Liquid at drain valve #10. Open valve #16 to
1:30	19	95	275	280	285	275	280	0	4.0	14	-48	-100	-165	-7	-69	-118	495	0	cool filters. Bolt of lightning caused power failure.
2:00	19	93	285	280	295	285	293	0	3.5	15	-46	-101	-168	-4	-51	-107	490	0	Start closing valve #32.
2:30	19	94	285	275	290	280	290	0	3.5	22	-42	-85	-166	-5	-64	-128	480	0	Change cold legs; rainy day, air moisture high.
3:00	19	91	290	285	300	285	300	0	3.5	21	-44	-102	-166	-8	-64	-112	480	0	
3:30	19	90	275	270	280	275	280	0	5	17	-46	-106	-166	0	-175	-112	500	5.2	Liquid in the kettle!
4:00	19	91	260	255	270	260	270	0	5	17	-46	-102	-162	0	-178	-109	500	0	Liquid level gage fluctuating violently.
4:40	19	91	275	270	295	275	285	0	5	16	-46	-99	-162	+5	-181	-106	500	0	
5:05				270															Start transfer of liquid.
6:00	20	91	275	292	290	270	290	0	7	17	-53	-99	-164	+12	-186	-106	440	0	
6:30	19	91	275	270	285	270	285	0	5	18	-48	-100	-165	+5	-186	-106	470	0	Shut down 6:35. 45 pounds air in can.